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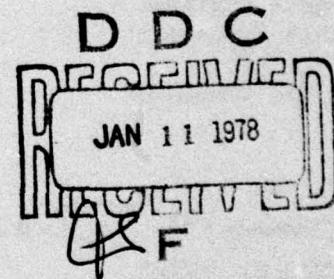
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## INVESTIGATION OF SEPARATION-LUBRICATION SUBSTANCES FOR USE IN ISOTHERMAL FORGING

WESTINGHOUSE ELECTRIC CORPORATION  
ADVANCED ENERGY SYSTEMS DIVISION  
PITTSBURGH, PENNSYLVANIA 15236

APRIL 1977



TECHNICAL REPORT AFML-TR-77-86  
Final Report for Period 16 December 1974 to 1 July 1976

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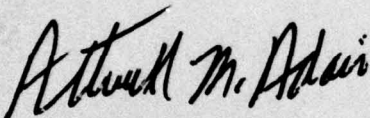


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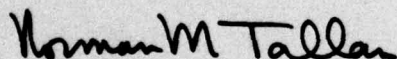
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This technical report has been reviewed and is approved.



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FOR THE COMMANDER



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19. KEY WORDS (Continue on reverse side if necessary and identify by block number) Isothermal forging, forging lubrication, glass lubrication, die-part adhesion, forging separation, titanium forging.	20. ABSTRACT (Continue on reverse side if necessary and identify by block number) A successful interface separation-lubrication substance for titanium alloy isothermal forging must simultaneously satisfy a number of demanding require- ments including: 1) elevated temperature stability in a preheating environment; 2) complete chemical compatibility with workpiece alloys and dies; 3) freedom from die accumulation tendencies; 4) minimal adhesion to the hot dies after forging; and	DDC RECEIVED JAN 11 1978 F	

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## 5) excellent friction reducing properties.

Evaluation techniques for these factors (except for friction reduction) were developed and applied to a series of separation-lubrication compositions representing both current state-of-technology materials and advanced formulations. Other factors such as ease of application and fusion behavior were determined. Several classes of advanced compositions were evaluated at specific temperature intervals from 1300F to 1800F. These systems included both workpiece pre-coat and die applied coatings. Die materials represented IN-100, TRW VIA Mod R and silicon nitride; preheat time ranged from one-half to four hours; and titanium alloys included both the alpha-beta Ti-6Al-4V and the beta stabilized Beta C<sup>+</sup>.

Advanced coatings representative of several new concepts of "vitreous binder plus particulate phase" formulations were supplied to Westinghouse for friction factor determination of ring compression testing. The new concepts included compositions containing boundary film additives, nucleating agents, and semi-abrasive particles. Die applied coatings evaluated were plasma sprayed stabilized zirconia, nickel aluminide alloy, and a reaction bonded silicon coating.



## FOREWORD

This report was prepared by TRW, Inc., Cleveland, Ohio under Westinghouse Electric Corporation Order Number 59-FZK-31010. The overall effort is included under USAF Contract Number F33615-74-C-5059 with Mr. F. J. Gurney as Westinghouse principal investigator. The Air Force Contract was initiated under Project Number 7351, "Metallic Materials", Task Number 735108, "Processing of Metals". The Air Force Contract was administered under the direction of the Metals and Ceramics Division, Air Force Materials Laboratory, Wright-Patterson Air Force Base, Ohio with Mr. A. M. Adair (AFML/LLM) as the Air Force Project Engineer.

This report covers work performed from December 16, 1974 to 1 July 1976 at TRW, Inc., Cleveland, Ohio. Dr. W. D. Spiegelberg of TRW, Inc. was the Project Engineer and Mr. C. R. Cook was the Program Manager. The work was performed at TRW, Inc. under the direction of Dr. I. J. Toth. Additional technical assistance on the program was provided by Mr. E. Thomas and Mr. A. Banones.

This report was submitted by the author on August 9, 1976.

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## I. INTRODUCTION

In hot die or isothermal forging processes, dies made from high temperature alloys are maintained at or near the desired working temperature. In current titanium alloy hot die forging practice this die temperature is generally maintained between 1600°F to 1700°F, but in the near future this temperature regime will be broadened. Utilization of advanced, beta stabilized titanium alloys will enable the use of lower die temperatures, and the range will extend downward to as low as 1300°F. With improvements in die systems and materials, the upper limits of the temperature range will be extended toward 1800°F particularly for the creep resistant alloys such as the near-alpha Ti 8Al-1V-1Mo or the alpha-beta Ti-6Al-2Sn-4Zr-2Mo.

The hot die forging process offers a capability for producing thin precise forgings to net or near-net dimensions. This can provide a major cost saving, particularly in aircraft structural components where material utilization factors are low and expensive machining operations are frequently used in conventional production.

A vital ingredient in the success of the hot die isothermal approach is a lubrication-separation substance that acts both to minimize friction and to separate the part from the die after the large scale plastic deformation that occurs during a forging operation for a part of complex configuration. Compounds employed in the past have been simple vitreous ceramics that provide excellent lubrication by hydrodynamic means but provide little or no assistance with separation.

In conventional forging practice, simple Newtonian fluids such as glass forming oxide compounds with viscosities in the range of 200 to 2000 poise at the forging temperature have been used as lubrication-protection media. In all cases, however, a thin film of graphitic or other type ceramic particulate material has been applied to the dies as a parting compound. This technique is successful so long as the die temperature remains sufficiently low that good adherence properties can be obtained and that graphite oxidation proceeds at a rate slow enough that it will provide the desired action.

In hot die forging, the use of such parting compounds has not proven practical except in those systems wherein the dies are cooled to ambient temperature between each forging. In these systems, application of, for example, a boron nitride powder with an inorganic binder can be reasonably effective. Similar formulations have not proven reliable for application to dies at elevated temperature and the process of cooling the dies between each forging is economically restricted to a fairly narrow range of part configurations and alloys.

Therefore, this program was initiated with the objective of developing commercially acceptable substances to be applied as coatings either to the workpiece or to the dies and meeting as a minimum the following requirements:



- 1) Workpiece coatings must be stable under elevated temperature exposure for periods of up to several hours in a preheating furnace prior to isothermal forging,
- 2) They must not contain ingredients either as trace elements or as intentional additions that can react with nickel alloy die materials during elevated temperature exposures that may extend to hundreds of hours (a thin film of lubricant resides at least at a few locations on the dies at all times during forging as well as during idling),
- 3) The coatings must not accumulate heavily in die cavity extremities such as ribs or projections in amounts sufficient to cause "lack-of-fill" appearances;
- 4) The lubricant-separation substances must assist in release from hot dies to minimize force requirements to minimize the likelihood of part distortion on ejection, and
- 5) The coating compositions must reduce friction to a minimum level so as to keep forging load requirements and localized die stresses to their lowest practical values.

These requirements together with more general considerations such as ease of application, environmental inertness, and cost were the subject of this investigation.

This report describes the laboratory formulation, quantitative characterization and final selection of compounds for isothermal forging over the potential near-term temperature regime.



## II. MATERIALS AND FORMULATIONS

This section is concerned with the raw materials that were utilized in the development of advanced separation-lubrication substances, with the rationale behind the selection of these materials, with the characterization performed to insure reproducible results, and with the formulation of the raw materials into successful workpiece or die applied coatings.

### A. Materials Selection

As discussed previously, some experience has been gained in prototype production isothermal forging work that has provided insight into the behavior of several different coating systems. The successes and shortcomings of these compositions have been described in a number of recently published reports.<sup>(1-6)</sup> Without exception, however, these reports documented an urgent need for improvement of several critical aspects of interface substances. In a recent program,<sup>(7)</sup> a lubricant development phase resulted in the successful formulation and field evaluation of a substance designated "OPT 112". This coating has proven superior for isothermal forging of large structural components where essentially infinite 1750°F preheat stability was a dominating requirement. This report describes a program that had as part of its scope the two-fold task of broadening the temperature regime of isothermal forging coatings into areas where OPT 112 is not necessarily the best choice, and optimizing the coating properties to improve where possible the stability, compatibility, adhesion, accumulation and friction properties beyond those available with the OPT 112 formulation.

To accomplish these tasks, materials were selected and combined as listed in Tables I to arrive at the isothermal forging compositions described in the following paragraphs.

#### 1. Vitreous Components

The majority of the coatings evaluated consisted of a vitreous phase to which was added "mill addition" components that acted by various means to reduce the adherence of the coating to the die while maintaining adherence to the titanium alloy. Compositions of the vitreous phase materials are listed in Table I with oxide concentrations listed in terms of weight percent in the matrix phase. The matrix materials listed in the table represent a selection from a screening effort performed in an early phase of the program to determine representative compositions that "fused" to a uniform glassy film on titanium coupons at the temperature increments of interest from 1300°F to 1800°F. Emphasis was placed on alkali free borosilicates in view of the die material compatibility limitations attributed in prior work<sup>(7)</sup> to the presence of sodium. Some attention was directed toward the glass forming boron oxide and boric acid materials without additions in view of their low cost and good viscosity characteristics.

All vitreous material was formulated to be suspended in an aqueous or organic carrier, applied as a slurry to the titanium workpiece and bonded in the green state with an organic resin that burned completely without char or other residue at a temperature below the softening point of the glasses.

TABLE I  
COMPOSITION OF COATING,  
WORKPIECE AND DIE MATERIALS

I. VITREOUS PHASE (WEIGHT PERCENT)

<u>22A</u>	<u>32B</u>	<u>38 BB</u>	<u>44A</u>	<u>46A</u>
42 SiO <sub>2</sub>	Markal	60 B <sub>2</sub> O <sub>3</sub>	100% B <sub>2</sub> O <sub>3</sub>	100% H <sub>3</sub> BO <sub>3</sub>
2 Na <sub>2</sub> O	Co.	31 SiO <sub>2</sub>	Crystal	Crystal
6 K <sub>2</sub> O	CRN	7 K <sub>2</sub> O		
49 PbO		2 CoO		
1 Li <sub>2</sub> O				
	<u>GNA</u> <u>46A</u>	<u>GCO</u> <u>48B</u>	<u>GFE</u> <u>54B</u>	
	67 SiO <sub>2</sub>	67 B <sub>2</sub> O <sub>3</sub>	80 B <sub>2</sub> O <sub>3</sub>	
	2 Al <sub>2</sub> O <sub>3</sub>	31 SiO <sub>2</sub>	19 SiO <sub>2</sub>	
	24 B <sub>2</sub> O <sub>3</sub>	2 CoO	1 FeO	
	7 Na <sub>2</sub> O			

II. PARTICULATE PHASE

A. Lamellar Solids

<u>Material</u>	<u>Code</u>	<u>Composition</u>	<u>Particle Size</u>
Bentonite	Thixo-Jel	67 SiO <sub>2</sub> , 16 Al <sub>2</sub> O <sub>3</sub> , 2 MgO 3 CaO, 3 Fe <sub>2</sub> O <sub>3</sub> , 3 Na <sub>2</sub> O, 0.4 K <sub>2</sub> O	-200 Mesh
Mica	No. 12 AlSiBronz	H <sub>2</sub> KAl <sub>3</sub> (SiO <sub>4</sub> ) <sub>3</sub>	-325 Mesh
Boron Nitride	BN 129	BN	0.03-0.7 Micron Grade
Graphite	#1651	C	-200 Mesh



TABLE I (CONTINUED)  
COMPOSITION OF COATING,  
WORKPIECE AND DIE MATERIALS

**B. Nucleating Agents**

Titanium Dioxide			
Rutile	TI 279	TiO <sub>2</sub>	1-5 Micron
Chem Grade	T 315	TiO <sub>2</sub>	" "
Cerium Oxide	CE 363	CeO <sub>2</sub>	-325 Mesh

**C. Semi-Abrasive Particles**

Titanium	Carbide	TiC	TI 276	-325 Mesh
----------	---------	-----	--------	-----------

**III. COMMERCIAL LUBRICANTS**

Deltaglaze	347M	Acheson Colloids
Deltaglaze	69	" "
Ceramgard	CG44	A. O. Smith
	CRT	Markal Corp.
	CRTHA	" "
	CRN	" "

**IV. TITANIUM ALLOYS**

**A. Titanium-6Al-4V**

			<u>C</u>	<u>Fe</u>	<u>N<sub>2</sub></u>	<u>Al</u>	<u>V</u>	<u>H<sub>2</sub></u>	<u>O<sub>2</sub></u>
0.040"	Sheet	AMS 4911B	0.02	.17	.009	6.5	4.4	73 ppm	.142
0.250"	Rod	" 4911B	0.026	.14	.021	5.98	4.12	0.009	.14
1.75" Diam	Bar	" 4928G	0.02	.21	.010	6.4	4.4	0.0077	.19

Sheet	RMI Heat	303927
Rod	RMI Heat	302452
Bar	Crucible Heat	G95041



TABLE I (CONTINUED)  
COMPOSITION OF COATING,  
WORKPIECE AND DIE MATERIALS

B. Titanium Alloy 38644 - Beta C

		<u>C</u>	<u>N</u>	<u>Fe</u>	<u>Al</u>	<u>V</u>	<u>Cr</u>	<u>Zr</u>	<u>Mo</u>	<u>O<sub>2</sub></u>
0.106	Sheet	.02	.014	.06	3.4	8.2	5.9	3.7	4.1	.104
2.75" Diam	Bar	.02	.010	.07	3.4	8.2	6.0	3.9	4.4	.097
0.460"	Plate	.02	.014	.06	3.4	8.2	5.9	3.7	4.1	.104
	Sheet	RMI	Heat	600393		STA				
	Bar	"	"	690195		STA				
	Plate	"	"	600393		STA				

V. DIE MATERIALS

A. IN 100 - Cast Bar & Plate

<u>C</u>	<u>Cr</u>	<u>Ni</u>	<u>Co</u>	<u>Mo</u>	<u>Ti</u>	<u>Al</u>	<u>B</u>	<u>Zr</u>	<u>V</u>
0.18	10.0	Bal	15.0	3.0	4.7	5.5	0.014	0.06	1.0

B. TRW VIA Modification R\* - Cast Bar & Plate

	<u>C</u>	<u>Co</u>	<u>Zr</u>	<u>B</u>	<u>Cb</u>	<u>Cr</u>	<u>Hf</u>	<u>Ta</u>	<u>W</u>	<u>Al</u>	<u>Ti</u>	<u>Mo</u>	<u>Ni</u>
Nominal	0.08	7.5	0.18	0.025	0.50	8.0	0.8	7.0	4.0	5.4	1.00	3.00	Bal.
Actual	0.09	7.4	0.17	0.030	0.49	8.2	0.7	7.1	4.3	5.7	1.01	3.05	Bal.

\*Experimental Rhenium-free version of TRW VIA

C. Silicon Nitride - Plate & Rod

Si<sub>3</sub>N<sub>4</sub> Hot pressed and sintered  
supplied by CERAC Inc.

TABLE I (CONTINUED)

COMPOSITION OF COATING,  
WORKPIECE AND DIE MATERIALS

VI. DIE APPLIED COATING MATERIALS

A. Zirconium Oxide

Plasma Spray Application

Metco 201B-NS ZrO<sub>2</sub>  
on TRW VIA

B. Nickel Aluminide

Plasma Spray Application

Metco 404 NS NiAl  
on TRW VIA

C. Silicon

Reaction Bonded

CG-11 and CG-38 supplied by A. O. Smith

Mixed with distilled water @ 3:12:20 ratio.

Applied to TRW VIA coupons.

Heated in hydrogen to 2000°F.



The fact that glassy, hydrodynamic lubrication alone is insufficient is shown by recent hot die forging experience with glassy coatings that lowered friction substantially but adhered tightly to both workpiece and dies forming tenacious glass "stringers" on removing the part from the die. This effect can be circumvented by the addition of particulates that interrupt the continuity of the film while retaining the excellent friction reducing characteristics of the vitreous matrix.

## 2. Solid Film Particulates

In order to overcome the disadvantages of the purely hydrodynamic lubrication mechanisms afforded by glasses and other mixed oxide systems, it has been found advantageous to add solid particles to the formulation to provide separating properties. These particulates preferably should have good lubricating tendencies of their own and thus the family of layered structure solids are likely to be beneficial. The particles themselves must not react with the binder component, must be at least partially wetted by the vitreous phase and must be stable both against decomposition and against oxidation in the atmosphere of the preheat furnace. Several candidate lamellar structured materials are discussed in the following paragraphs.

### a. Graphite

The most commonly utilized lubricating substance for hot working applications is electric furnace graphite. Part a of Figure 1 is a scanning electron microscope photograph of a sample of lubricating graphite in flake form. The particles are relatively fine with a "B" type particle size distribution. This material clearly exhibits the easily sheared structure that consists microscopically of adjacent flat planes of ionically bonded carbon atoms separated by weak Van der Waals bonds between the planes. Graphite is inexpensive, has demonstrated excellent qualities in reducing friction, is available in a variety of particle size classifications and has demonstrated success in prior isothermal forging experience. The major impediment toward its use is its tendency toward oxidation depletion with extended preheating at temperatures of 1300°F and above. However, for low temperature preheating for relatively short times of exposure, its utilization was considered.

### b. Boron Nitride

Figure 1 illustrates the very similar structural configuration of boron nitride (chemical formula BN). This material exhibits all the attributes of graphite that lead to success in isothermal forging, including a similar lamellar structure, weak bonding between layers and an availability in controlled particle size grades. In addition, it exhibits a singular characteristic of being inert to chemical reaction with both oxygen and with vitreous oxides over the temperature range from 1300-1800°F. A disadvantage of BN is its cost, which exceeds 100 times that of an equivalent grade of graphite. Also, most commercial lubricant manufacturers do not supply boron nitride colloids for lubrication purposes because of this cost. This situation may well change as isothermal forging of titanium alloys reaches increased commercial importance.



a. Graphite

5000X



b. Boron Nitride

5000X

Figure 1. Scanning Electron Microscope Photo of Two Types of Lubricant Particles.



c. Mica, Talc and Clays

Layered structures are exhibited also by the complex silicates of the elements aluminum, magnesium, calcium, sodium and potassium. Kingery<sup>(8)</sup> materials have been suggested as forging lubricant additives for conventional hot working operations. Mica<sup>(9)</sup> and talc<sup>(10)</sup> have been the subject of patents in the lubricant area and clays<sup>(11)</sup> including bentonite, kaolinite and montmorillonite have demonstrated success, although usually in combination with other materials. Bentonite is a common ingredient in minor amounts in graphitic die lubricants. In this application it acts as a stabilizing agent, acting to prevent graphite particles from settling out of suspension. The materials are inexpensive and readily available.

d. Nucleating Agents and Chemical Modifiers

The conditions existing in a thin glass film on a titanium alloy workpiece are not necessarily those of chemical equilibrium. Temperatures are low and exposure times are short and kinetics rather than thermodynamics governs the material behavior.

Under these conditions the addition of finely divided particles of a nucleating agent, such as  $TiO_2$  or a chemical modifier such as  $CeO_2$  can assist in weakening the affinity of the coating for the nickel alloy die.

d. Other Oxides and "Hard" Particulates

Many materials not possessing a lubricating characteristic through a lamellar structure can be obtained inexpensively in very fine particle sizes and demonstrate chemical inertness and thermal stability. Several of these are common abrasive materials such as carbides, nitrides and refractory oxides. While not specifically enhancing frictional characteristics, these ingredients may substantially enhance separation qualities of lubricant precoat. An important consideration in the use of these is their size as they must be small enough and suitably dispersed to avoid deleterious surface impressions in forgings or abrasion on hot dies.

B. Workpiece, Die and Die Coating Materials

Other materials utilized in the program are also listed in Table I.

Two titanium alloys were evaluated, including the alpha-beta type Ti-6Al-4V, and the deep hardening Beta C (Ti 38644) alloy. Die materials included cast IN-100 and an ultra-high creep strength nickel base alloy TRW VIA, considered a candidate die material for future high temperature applications. A ceramic die insert material, silicon nitride, was also utilized.

The die coating work was directed primarily toward three processes, plasma sprayed stabilized zirconia, plasma sprayed Ni-Al, and a reaction bonded silicon coating.

### III. EVALUATION PROCEDURES

The following describes the rationale and procedures involved in the evaluation of separation-lubrication substances in both the initial effort concerned with current state of the art substances (Section IV) and in the later work concerned with improved varieties. It is important to recognize that throughout this program emphasis was placed on quantitative characterization of coating properties to allow accurate comparison between substances, to provide a measure of the rate of convergence toward pre-established goals and to determine the correlation between formulation level evaluation and commercial practice.

#### A. Visual Stability and Cohesion

A visual stability evaluation was performed on each of the state of the art formulations and an occasional sample from the advanced formulation phase. This procedure determined the response of a coating to elevated temperature in terms of its thickness change and continuity.

The procedure was as follows:

Titanium alloy (Ti-6Al-4V) rods nominally 1/4 inch in diameter by four inches long were coated over a two-inch length in the rod center. Coating was performed by immersion with masking applied to the free ends of the samples. The coating thickness was measured on a 10 power optical comparator at five stations along the length of the coated zone and at one position on the uncoated bar. Following these thickness measurements, the bars were suspended horizontally and tightly clamped over a 3/4-inch length at each end in watercooled copper grips. A platinum-platinum-10% rhodium thermocouple was spotwelded to a point on the rod previously determined to be the center of the hot zone in the rod (not necessarily geometrically centered between the grips because of differences in water cooling efficiency between the ends). This thermocouple acted both to control and to monitor temperature precisely through the feedback control system of the apparatus. A small region directly adjacent to the thermocouple weld was scraped bare of coating because long time exposure to silaceous glassy oxides causes degradation of the platinum and loss of weld integrity.

Photographs of the samples were taken at hourly intervals during the three-hour 1750°F exposures. During the photograph the samples were illuminated with a 250 watt reflector flood lamp. This lighting revealed the condition of the coating surface without complication caused by light transmitted from the glowing titanium alloy rod.

The samples were brought to temperature at a controlled gradual rate reaching 1750°F in three minutes. This procedure approximately simulated the heatup condition in a preheat furnace and also allowed a visual determination of significant character or color changes in the coating with increasing temperature.



## B. Thermogravimetric Analysis - Weight Stability

A sequence of measurements determined the weight changes encountered by coatings during exposure in the oxidizing atmosphere of an electric resistance furnace. Several objectives were sought. First, a weight gain at high temperature (e.g., 1700°F) indicates a lack of protection by the coating. Second, a weight loss at low temperatures (e.g., 600°F) indicates burning of organic binders and other volatile lubricant components. Third, and most important, weight changes at intermediate temperatures (e.g., 1300°F) indicate the progress of chemical reactions within the coating itself such as oxidation or degradation of inorganic binder or particulate phase material. The intermediate temperature was chosen as 1300°F since alpha case formation on coated titanium alloy samples does not represent a significant weight change mechanism.

The procedure was as follows:

The lubricant test coupons were cut from Ti-6Al-4V sheet nominally 0.042 inches thick. Samples were sheared from a single sheet into squares 1.010<sup>±</sup>.010 inches on each side. These were subsequently clamped together and surface ground in the edgewise direction to 1.000<sup>±</sup>.002 inches in order that all samples should be virtually identical in surface areas. Following surface grinding a hole was drilled through the flat faces of the coupons near one corner. This hole was drilled to accept a metal wire hanger for immersion coating and exposure.

A rack for holding samples during high temperature exposure was fabricated from stainless steel rods affixed to a high density magnesite firebrick. Lubricant test coupons were hung on this rack, one sample of each lubricant for each exposure.

Following furnace exposure the samples were removed from the hangers, cooled slowly and then placed in individual weighing cups and weighed on an electronic balance. Coupon thickness was determined before and after coating with a dial indicating micrometer with a spring loaded anvil preset to apply a 40 gram load. Thickness readings were taken at four positions on the pieces and averaged to obtain the values reported.

## C. Coating - Die Material Compatibility

The literature describes three types of oxidation-corrosion mechanisms pertaining to nickel-base alloys operating at medium to high temperatures. One, which has been termed "green rot,"<sup>(12)</sup> is associated with a carburizing or a cyclic oxidizing-reducing environment acting to continually deplete surface and subsurface parent metal of chromium. A second<sup>(13)</sup> involves continual solution of the normally protective oxides by low melting oxides which are liquid (e.g., MoO<sub>3</sub>, V<sub>2</sub>O<sub>5</sub>) at the service temperature. A third, usually called "sulfidation corrosion,"<sup>(14)</sup> requires the presence of small quantities of sodium and sulfur in an oxidizing environment and, once initiated, can proceed autocatalytically.

Die corrosion in isothermal forging need not be catastrophic to be problematical. Even a minor degree of pitting or surface fouling can interfere with production operation. A few cases of catastrophic corrosion have been encountered in commercial hot die forging practice and it is extremely important

to avoid future reoccurrences. Equally important, however, is a need to recognize that such corrosion is likely to be a significant component of die wear on expensive superalloy dies and lubricant formulations should be tailored where possible toward maximized compatibility with advanced die materials.

The procedure followed in this program's evaluation of coating-die material compatibility is as follows:

Cylindrical samples approximately 0.400 inches in diameter by 0.400 inches high were abrasive sectioned from bars of investment cast IN-100 or TRW VIA. Following sectioning, both faces of each sample were ground flat and parallel to within 0.001 inch and then a conically tapered-flat bottomed recess, 0.080 inches deep by 0.340 inches in diameter was machined in the "upper" face. A selection was made from those machined to include those most closely matching in weight and these were used as a set for elevated temperature exposure.

The samples were exposed for sixteen hour periods at 1650°F in an electric resistance air atmosphere furnace. After each exposure the samples were removed from the furnace on an Inconel tray and cooled to room temperature. Visual observations of the coating condition were recorded and then the lubricant was removed from the slugs by blasting with fine zircon sand (on the coated face only) for a time sufficient to remove all the fused material. This time measured approximately five to ten seconds for each coating, and varied with coating "hardness".

Two control samples were carried throughout the procedure, one being exposed in the furnace with no lubricant applied while the other was not exposed but was zirc blasted each time in a manner similar to the exposed pieces. The former control sample provided an estimate of the oxidation weight loss while the latter control sample provided a measure of the blasting loss.

Samples were weighed on an Ainsworth precision electronic balance to an accuracy of  $\pm 0.5$  milligram after each exposure and cleaning, and sample heights were measured with a pin micrometer. The total solid content of the lubricant applied to each slug was maintained constant for each lubricant and each application at  $100 \pm 5$  milligrams.

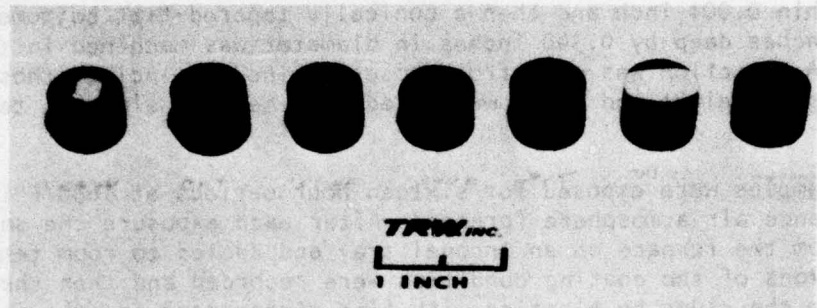
A photograph of the set of six IN-100 slugs immediately following a 16-hour exposure is provided in Figure 2. The exposure shown was taken on an initial trial compatibility sequence before the machined recess was provided. The photo thus shows the tendency for the Acheson formulations to spread over the sides of the slugs. Provision of the recess resulted in a more uniform exposure condition and reduced variability in the weight loss data.



to avoid future occurrences. Equally important, however, is a need to recognize that such corrosion is likely to be a significant component of the wear on expensive superalloy dies and lubricant formulations should be tailored where possible toward maximum compatibility with advanced die materials.

The procedure followed in this program's evaluation of coating-die material compatibility is as follows:

Cylindrical samples approximately 0.500 inches in diameter by 0.400 inches high were abrasive sectioned from both ends of investment cast IN-100 or Ti-6Al-4V. Following sectioning, both faces of each sample were ground flat and polished to within 0.0005 inches and then chemically etched for 10 seconds in 0.050 N Nital based on 0.5% nitric acid in dimethyl sulfoxide. The etched faces were then coated with a thin layer of gold for electron microscopy. The samples were then exposed to the furnace conditions for 16 hours at 1650°F. The samples were then sectioned and polished to within 0.0005 inches and then chemically etched for 10 seconds in 0.050 N Nital based on 0.5% nitric acid in dimethyl sulfoxide. The etched faces were then coated with a thin layer of gold for electron microscopy.



**Figure 2. Lubricant - IN-100 Compatibility Samples After 16 Hour Exposure in Air at 1650°F.**

exposed in the furnace with the lubricant applied. The samples were then sectioned and polished to within 0.0005 inches and then chemically etched for 10 seconds in 0.050 N Nital based on 0.5% nitric acid in dimethyl sulfoxide. The etched faces were then coated with a thin layer of gold for electron microscopy.

Samples were weighed on an Alpkem 1000 electronic balance to an accuracy of 0.2 milligrams after exposure and cleaning. The samples were then sectioned and polished to within 0.0005 inches and then chemically etched for 10 seconds in 0.050 N Nital based on 0.5% nitric acid in dimethyl sulfoxide. The etched faces were then coated with a thin layer of gold for electron microscopy.

A photograph of the set of six IN-100 dies immediately following a 16-hour exposure is provided in Figure 2. The exposure shown was taken on an electron microscope. The samples were then sectioned and polished to within 0.0005 inches and then chemically etched for 10 seconds in 0.050 N Nital based on 0.5% nitric acid in dimethyl sulfoxide. The etched faces were then coated with a thin layer of gold for electron microscopy.

#### D. Coating Adhesion and Accumulation Properties

Among the most vital properties required of isothermal forging lubricants are low rates of accumulation on dies and low release forces after completion of forging. An inability to date to achieve perfection in these properties limits the precision attainable with the isothermal process. In response to the need for a characterization of lubricant properties with regard to these requirements, it was decided within this program to develop techniques to measure properties directly related to accumulation and release.

In conventional titanium alloy forging processes where ceramic workpiece precoat are used on the part and die lubricants (usually graphitic) are applied to the die, accumulation and release are not problems limiting forging precision. The die coating compound in that case acts as a very efficient parting compound as well as a lubricant, thus preventing adherence of the ceramic precoat to the dies.

In isothermal forging, a suitable die coating has not been developed. In processes used to date the die precoat has come directly into contact with the hot dies. A certain "partition ratio" between the amount of precoat left on the dies and the amount retained on the part is attained. If the forging process is "isothermal" and if the lubricant wets both workpiece and die equally, then this partitioning will occur at approximately 50%, leaving about half the coating on the dies and carrying away half with the workpiece.

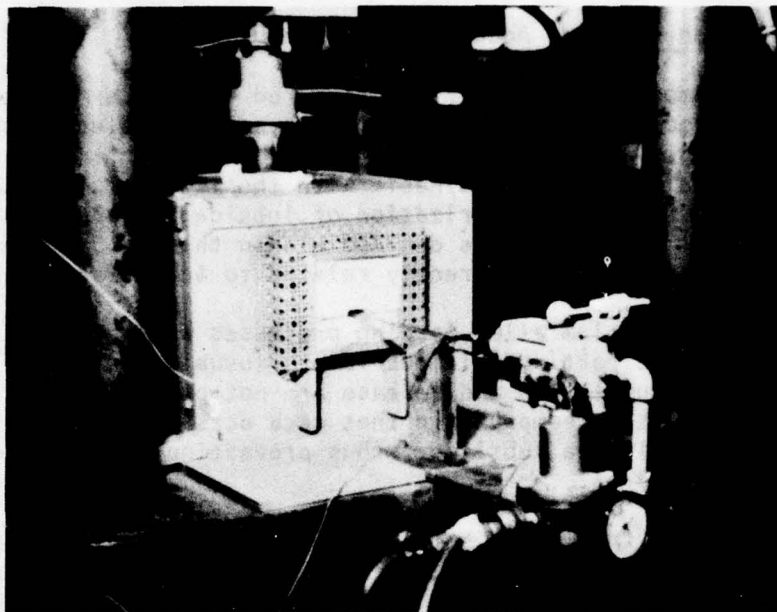
The following describes the apparatus, procedure and results of measurements of the adhesive characteristics of lubricants:

Figure 3 illustrates the external construction of the lubricant adhesion apparatus. Part a of the figure shows details of the radiant heated furnace mounted in a 10 ton Greenerd hydraulic press. The pneumatic actuated withdrawal mechanism including strain gage instrumented pull bar is shown in position between the withdrawal cylinder and the furnace. A linear variable displacement transducer is rigidly attached to the frame of the release lever and measures the relative amount and rate of sliding of the test samples located inside the furnace.

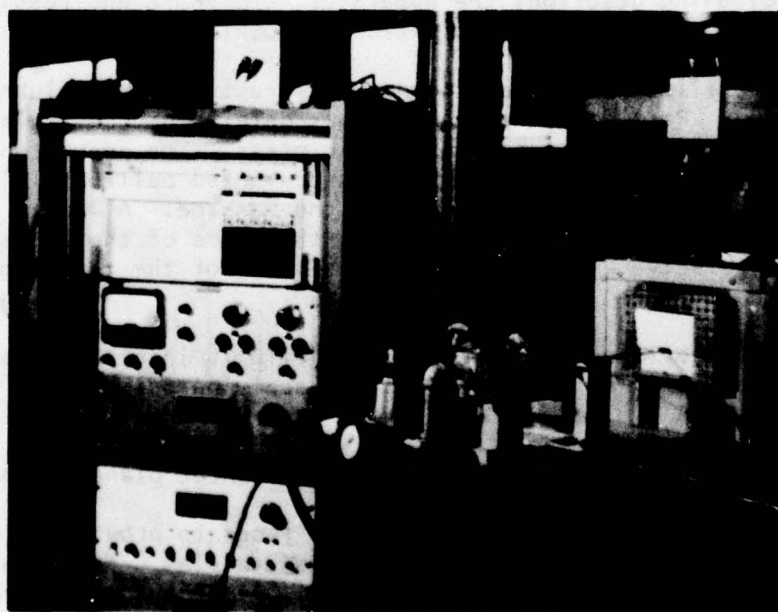
A moveable cylindrical bar made from U710 is inserted through the top of the furnace acting as the upper die for applying pressure to the lubricated samples. A similar U710 bar (both bars 2 1/4" diameter) anchored to, but insulated from the press bed provides the lower support die. The two bars meet at a position very close to the thermal center plane of the furnace.

Each rod is grooved on the mating end faces to provide lateral support and guidance for the 1-inch wide adhesion samples. The samples themselves are 1-inch wide by 3-inches long by 1/8-inch thick and are surface ground flat and parallel to within 0.001 inch on the flat surfaces. Holes drilled in one end of the samples receive mating dowel pins provided on the ends of pull bars, a stationary one affixed to the rear of the furnace and a movable one attached through the furnace door, across a support block and





a. Front View of Furnace and Withdrawal Mechanism



b. View of Furnace and Recording Instrumentation

**Figure 3.** Isothermal Forging Lubricant Adhesion Measurement Apparatus as Modified From ASTM D 905-72.

finally to the pneumatic actuating mechanism. A water-cooled load cell is in series with the upper load bar as seen in Figure 3a.

Figure 3 illustrates an alternate view of the apparatus and shows the light pen recording equipment that displays and records the test data. Parameters monitored on the oscillograph traces include: upper load cell output, pull bar extension, lower die temperature (from a thermocouple located within 1/8-inch of the samples) and LVDT output during testing.

The experimental procedure consists of the following:

- 1) Either one or both of the previously sandblasted samples are coated with lubricant by immersion or spraying to a precisely controlled thickness as measured by a dial indicating micrometer. The coating covers a one-inch section at the end of the sample opposite the hole.
- 2) The samples are inserted in the furnace previously stabilized at the desired temperature for several hours. The lower sample is located accurately on the inconel dowel pin anchored to the back of the furnace. The upper sample is then placed in position on an Inconel connecting link and attached to the instrumented strain bar.
- 3) After a dwell at temperature for sufficient time to bring the samples and lubricant to the furnace temperature, a load is applied by the hydraulic press nominally 3000 lb or 1-1/2 tons per square inch of contact since the samples overlap over an area measured as  $1.000 \pm 0.050$  square inches. This overlap area is measured following each test and is applied to the calculation of the shear stress and the applied tonnage.
- 4) The load is released after a 2-minute application period. This load magnitude and duration were selected from preliminary testing as conditions that cause a very small amount of plastic deformation in titanium alloy samples, but not enough deflection to wedge the pieces into the die channel.
- 5) Following load release, the lubricated samples are pulled apart in shear by actuation of the release mechanism. The air pressure is increased in small increments until a load is reached that will cause noticeable "creep" of the upper sample. This pressure is then maintained for the duration of the test.



#### IV. ASSESSMENT OF CURRENT TECHNOLOGY

This section summarizes the results of an effort devoted to an assessment of several coating systems that were utilized on recent isothermal forging programs to establish a baseline for the second phase involved with evaluation of alternative coating systems. Corresponding data tabulations for these systems are included in Appendix A.

The coating systems evaluated in the state-of-technology assessment were five and included the Markal Corporation products CRT and CRT-HA, Acheson Colloids D347M and D69 (also referred to as W0315 in a pre-commercial version) and the TRW formulation, OPT 112 developed under subcontract to Wyman-Gordon Company and AFML.

##### A. Spectrochemical Characterization

Lubricant samples were analyzed spectrochemically to determine levels of selected elements for correlation with lubricant-die material compatibility data. It has been shown in a previous investigation that a combination of sulfur and sodium represents a potential hazard from an elevated temperature sulfidation standpoint and that sodium alone can be detrimental, but the possibility that other elements may cause abnormal effects on die materials has not been discounted. Therefore, a semi-quantitative spectrochemical analysis covering virtually all elements detectable by this technique was conducted and the results are presented in Table A-1. A separate analysis by oxygen combustion was carried out for sulfur.

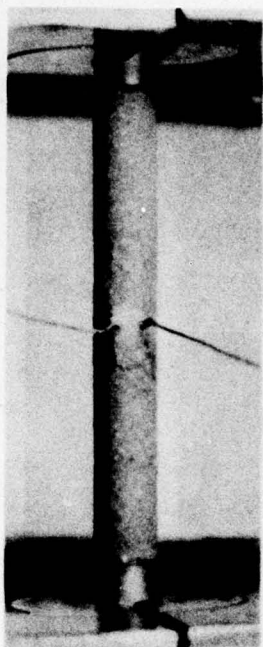
The table demonstrates the virtual absence of elements such as lead, antimony, bismuth, vanadium or mercury that may be harmful. Sodium was found at nominally minor levels in the Acheson formulations, and the sulfur level in OPT 112 was highest of the lubricants examined although still relatively low. It should be remembered, however, that neither sodium nor sulfur individually in these concentrations has been traced as the source of a corrosion problem and therefore all five compositions are considered acceptable.

##### B. Visual Stability and Cohesion

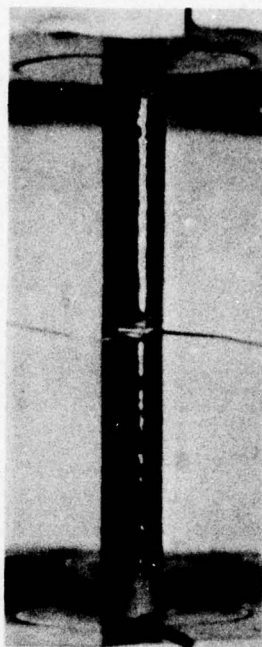
Photographs of two coating systems evaluated by this technique are illustrated in Figures 4 and 5. They reveal the glossy non-flowing character of CRT and the matte finish appearance of the boron nitride containing OPT 112.

Significant color or character changes noted during heating were as follows:

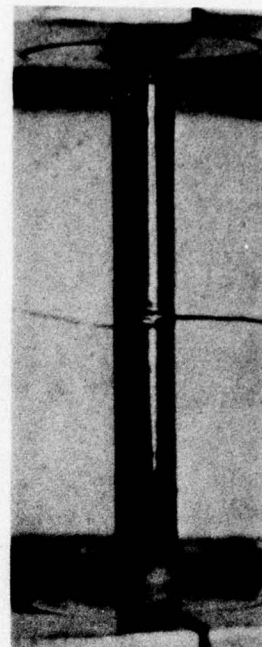
- 1) CRT changed abruptly from dark gray to white at 800°F. At 1500°F the coating fused and became glossy.
- 2) CRT-HA changed from dark gray to white also, but at 750°F. The coating fused to a wavy character at 1400°F, but the coating became smooth after a brief interval at 1750°F.



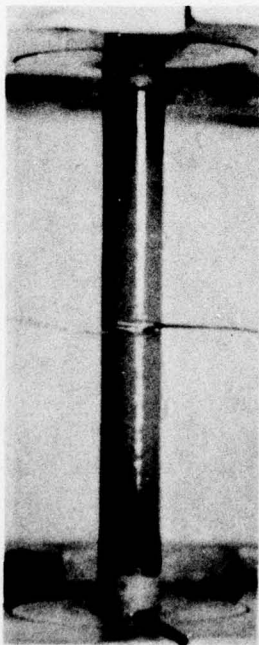
Room Temperature  
Before Thermal Cycle



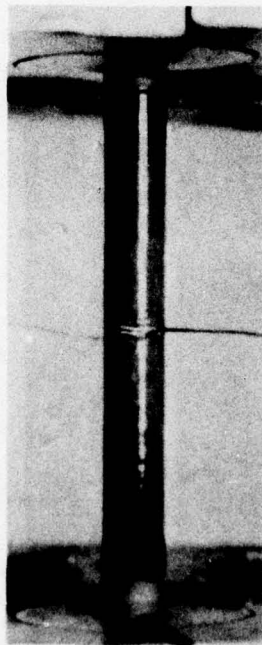
At 1750°F



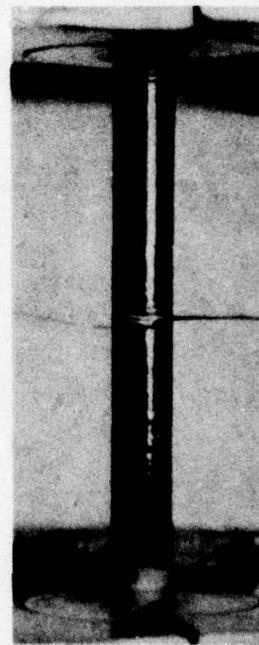
1 Hour at 1750°F



2 Hours at 1750°F



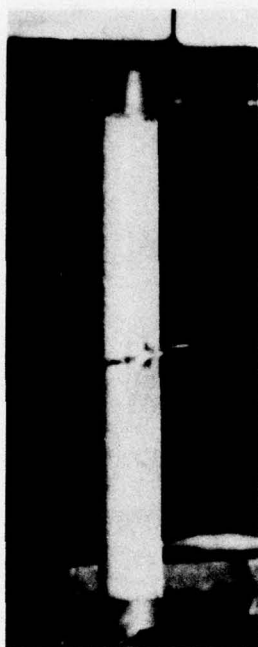
3 Hours at 1750°F



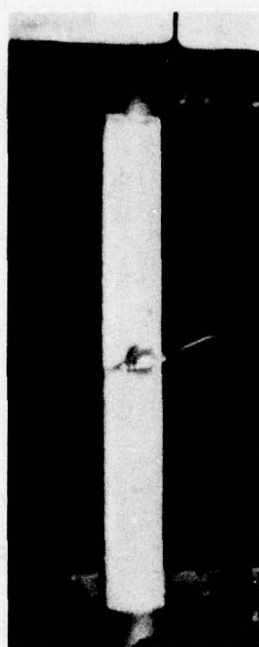
R.T. After 1750°F

Figure 4. Lubricant Visual Stability - CRT

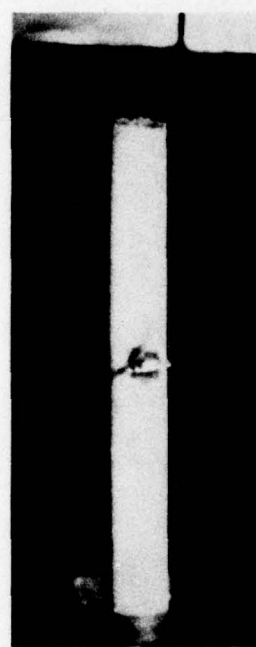




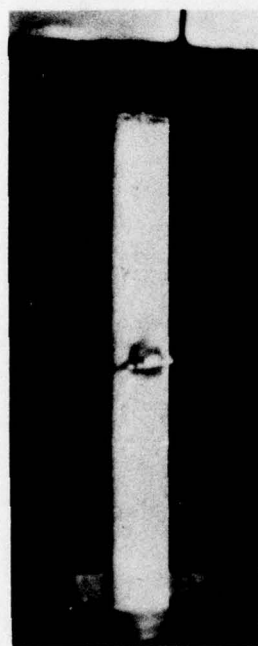
Room Temperature  
Before Thermal Cycle



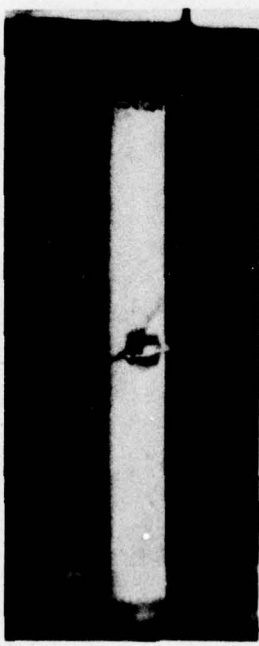
At 1750°F



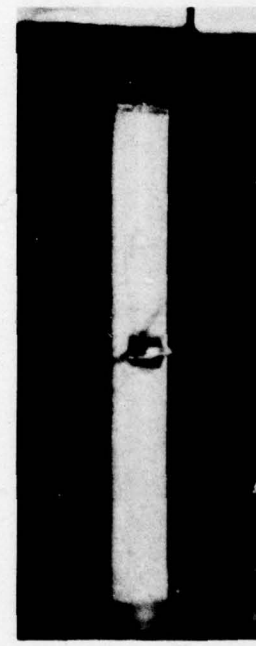
1 Hour at 1750°F



2 Hours at 1750°F



3 Hours at 1750°F



R.T. After 1750°F

Figure 5. Lubricant Visual Stability - OPT 112

- 3) D69 developed a pattern of cracks at 800°F. At 900°F there was an abrupt change from the normal green color to a light brown. Between 900 and 1140°F a gradual deepening of the color occurred. The coating became glassy after a hold period at 1750°F.
- 4) D347M changed abruptly from green to white at about 800°F. At 1070°F the coating took on a gray cast. Fusion took place after a dwell at 1750°F.
- 5) OPT 112 changed from gray to white at about 850°F. It maintained a matte white surface that showed a glassy reflective character on close examination. The exact point of fusion, however, is not obvious but lies in the range of 1500-1600°F.

Table A-II lists the coating thickness data for coatings applied by immersion on the rod samples. All coatings as applied were fairly uniform and exhibited a minimum amount of taper from end to end. The change in thickness before and after thermal exposure was also quite uniform and reproducible between samples and between lubricants.

#### C. Thermogravimetric Analysis

Table A-III summarizes the results of the weight and thickness determinations for the selected lubricants. The weight change data show that virtually all of the weight loss occurs during the 600°F exposure period and thus corresponds to removal of organic binders or other volatile material. Different binder fractions among formulations contributes to the observed differences at the low temperature exposure. At 1300°F, a slight decrease in weight was observed relative to the 600°F exposures, but the weight loss was not a function of time at temperature. This is the ideal behavior since it indicates coating stability from oxidation or chemical reaction. The exposures at 1700°F similarly show only slight weight changes for all lubricants indicating the protective nature of the coatings was sufficient at 1700°F for each lubricant.

Thickness readings for the lubricants are also recorded in Table A-III. These are averages taken at four positions. Some comments concerning the variability of the thickness measurements are worthy of noting here.

Before coating, the sheet thickness measurements were precise to within  $\pm 0.0001$  inches for all four readings taken on individual coupons. Some variation in sheet thickness from sample to sample was found, but this was small. The maximum thickness was 0.0430 inches and minimum was 0.0415 inches.

After coating and drying, considerably more variability was introduced into the thickness measurement, since the immersion technique results in a tapered film profile. The degree of taper depends on slurry viscosity and rate of solvent evaporation, which in turn is a function of coupon temperature at



time of application. This taper was found to vary among the lubricants with the largest value being 0.0019 inches per inch per side for W0 315 and smallest value being 0.0009 in/in/side for OPT 112. After exposure the taper was still present, and was accentuated in most cases by viscous flow especially for the 1700°F tests.

It is a well established fact that lubricant thickness is a critical variable in isothermal forging operations. Recognition of the additional fact that variations in coating thickness can occur as a result of slurry variables, preform temperature or solvent system characteristics is important if the full benefit of the precision capabilities of the isothermal forging process are to be achieved.

#### D. Coating Compatibility With IN-100 Die Material

The results of an accumulated exposure time totalling 176 hours are listed in Table A-IV. This table is in terms of cumulative weight loss, expressed as a percent of the original sample weight for each of the six lubricants tested and the two control samples. The visual appearance and color as-fused and cooled is also listed. The coated samples were given two additional exposures after the 176-hour tests to determine whether the attack continued with further heating as might be expected if an intergranular diffusion mechanism were active.

Figure 6 illustrates the rate of weight loss plotted against time for the six formulations. The data have been corrected for cleaning loss by subtracting the average weight loss of the control samples from the weight loss exhibited by the coated pieces. It is seen from the graph that the CRT and CRT-HA coated samples suffered the highest rate of weight loss while the D347M formulation showed the least. It is to be emphasized that in no case was the degree of attack considered serious. The data, however, provide a logical basis for comparison in subsequent experimental development and in specifying allowable levels of impurities in commercial practice.

#### E. Adhesion and Accumulation Properties

Adhesion measurements were made for a variety of conditions including titanium alloy against titanium alloy samples and titanium alloy against IN-100 samples. The complete record of data is recorded in Table A-V. In this table are recorded the adhesive shear strength measurements as corrected for the actual contact area, the degree of shear "creep" (in other words, the distance the samples slid relatively before the load dropped to zero), and the load applied prior to shear stress measurement. These were interpreted from the oscillograph traces. Lubricant thicknesses, heat cycle time and temperature and sequence numbers are also listed. The coated samples are designated with asterisks and sorted by upper and lower samples in contact.

The data are further broken down in Table II to show the effects of die material and lubricant thickness on adhesive strength. The values listed in the table are shear strengths (in psi) and reflect an average and standard deviation.

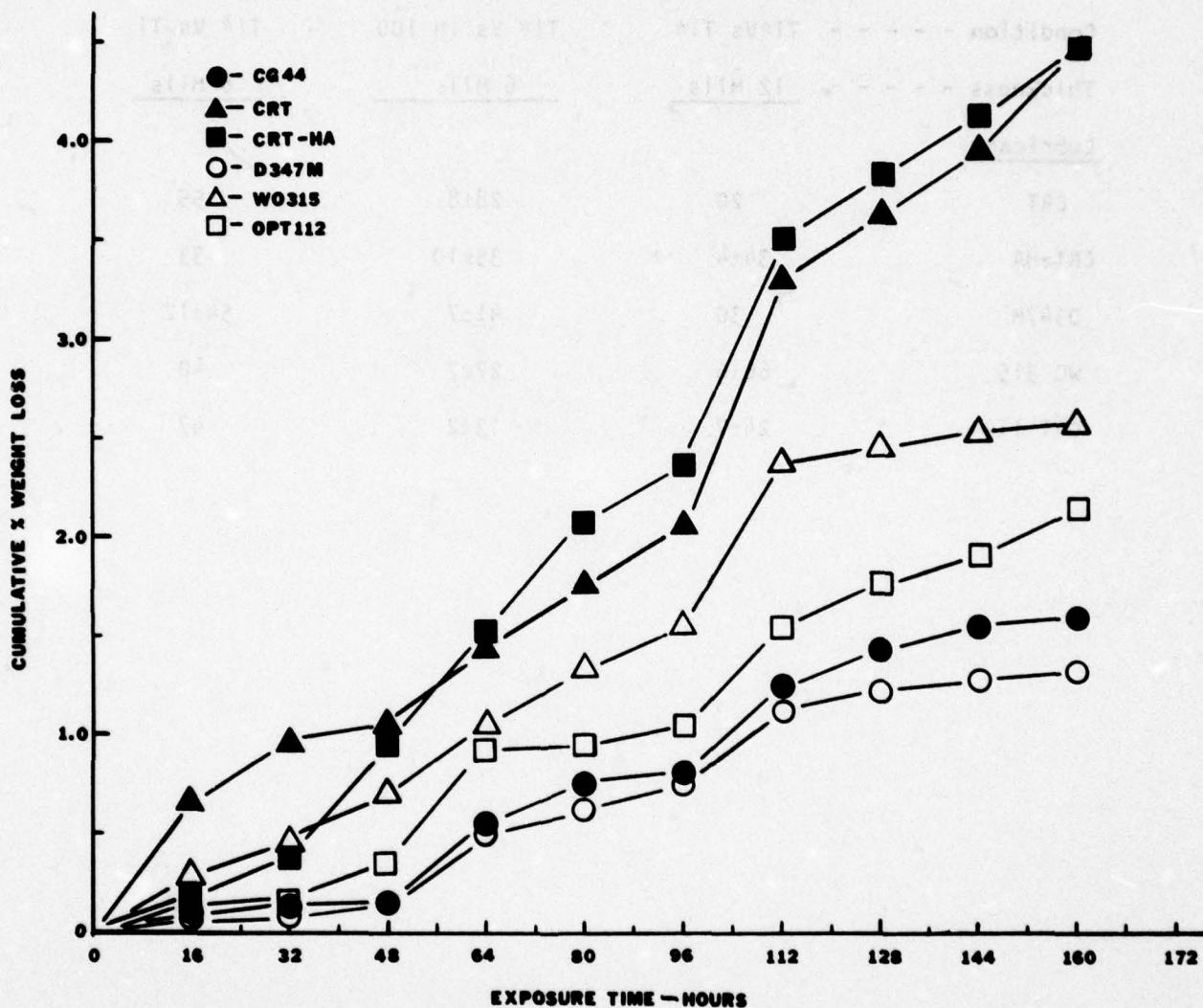


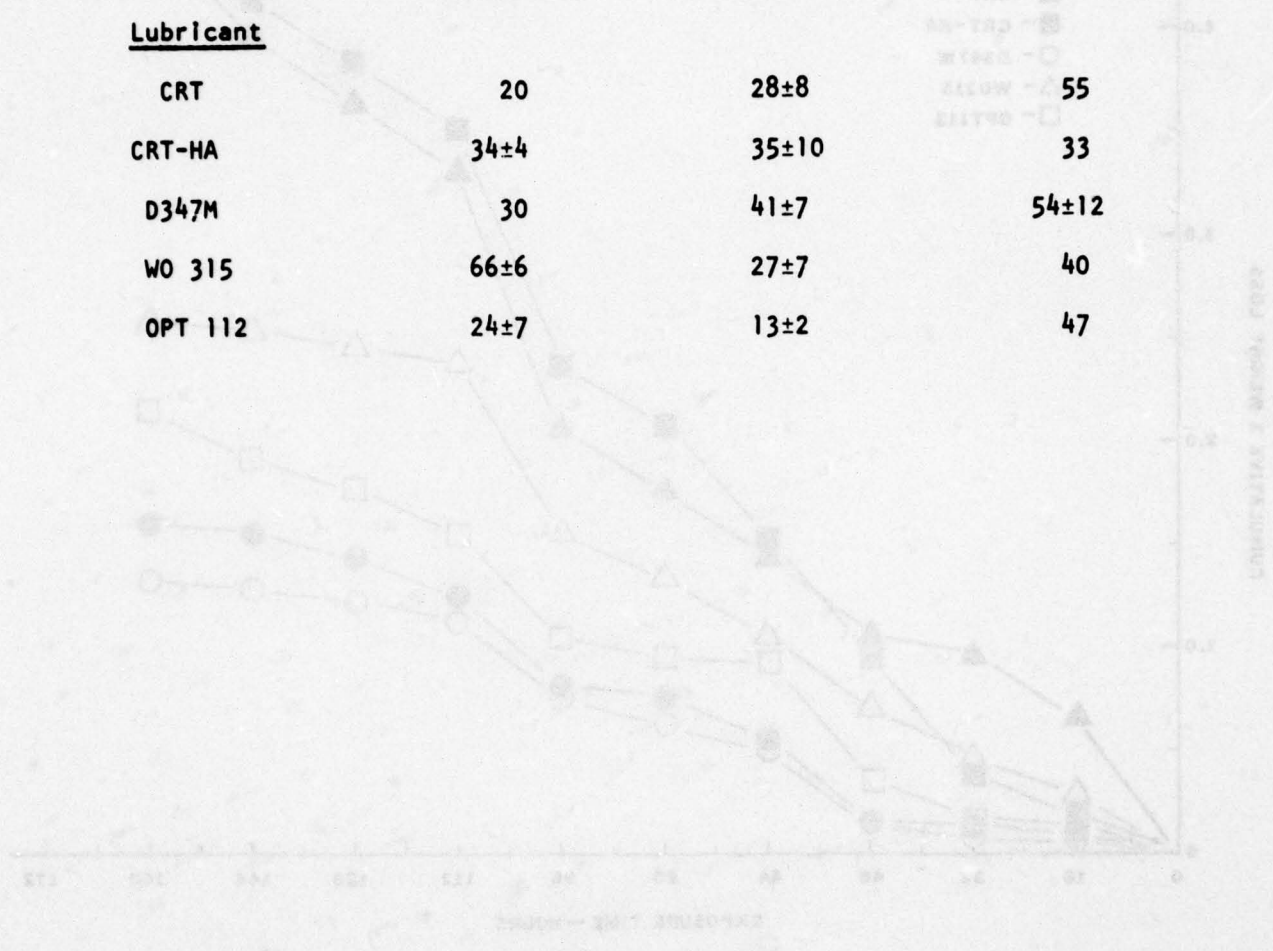
Figure 6. Cumulative Weight Loss vs. Exposure Time for Lubricant - IN-100  
Compatibility Corrected for Control Sample.



**TABLE II**

**ADHESION MEASUREMENT DATA - SUMMARY**

Condition - - - - -	Ti*Vs Ti*	Ti* Vs IN 100	Ti* Vs Ti
Thickness - - - - -	<u>12 Mils</u>	<u>6 Mils</u>	<u>6 Mils</u>
<u>Lubricant</u>			
CRT	20	28±8	55
CRT-HA	34±4	35±10	33
D347M	30	41±7	54±12
WO 315	66±6	27±7	40
OPT 112	24±7	13±2	47



The table exhibits several interesting features. First, the adhesive strengths exhibit a lubricant-to-lubricant variation that is large compared to the variation within tests. The CRT and CRT-HA formulations have generally higher values than the others apparently correlating with the high accumulation rates and difficult removal experienced in forging practice.

Second, a difference is apparent between the titanium alloy on titanium alloy values and the titanium alloy on IN-100 values. It is anticipated that these numbers also correlate with isothermal forging separation-lubrication quality from the release and accumulation standpoint.

Third, the standard deviations are in general larger than desirable but statistically significant tests can still be made. It is to be remembered that the data in Table A-V were obtained during an evolutionary process and future tests are expected to show better reproducibility. Nevertheless, at least three tests will be required to detect differences of as great as 20% with a reasonable confidence level.



## V. EVALUATION OF ADVANCED FORMULATIONS

A comprehensive program of selection and evaluation was successfully applied to a number of advanced compositions based on the lamellar solid, chemical modifier, and refractory particulate concepts described previously. The materials utilized in these are listed in Table I and the evaluation data are summarized in Appendix B. In the text that follows, some highlights of the results are presented with emphasis on application to both beta and alpha-beta titanium alloys, to cast nickel alloy die materials of the IN-100 and TRW VIA mod R types, to a potential ceramic die insert material,  $\text{Si}_3\text{N}_4$ , and to nickel alloy dies protectively coated by various techniques.

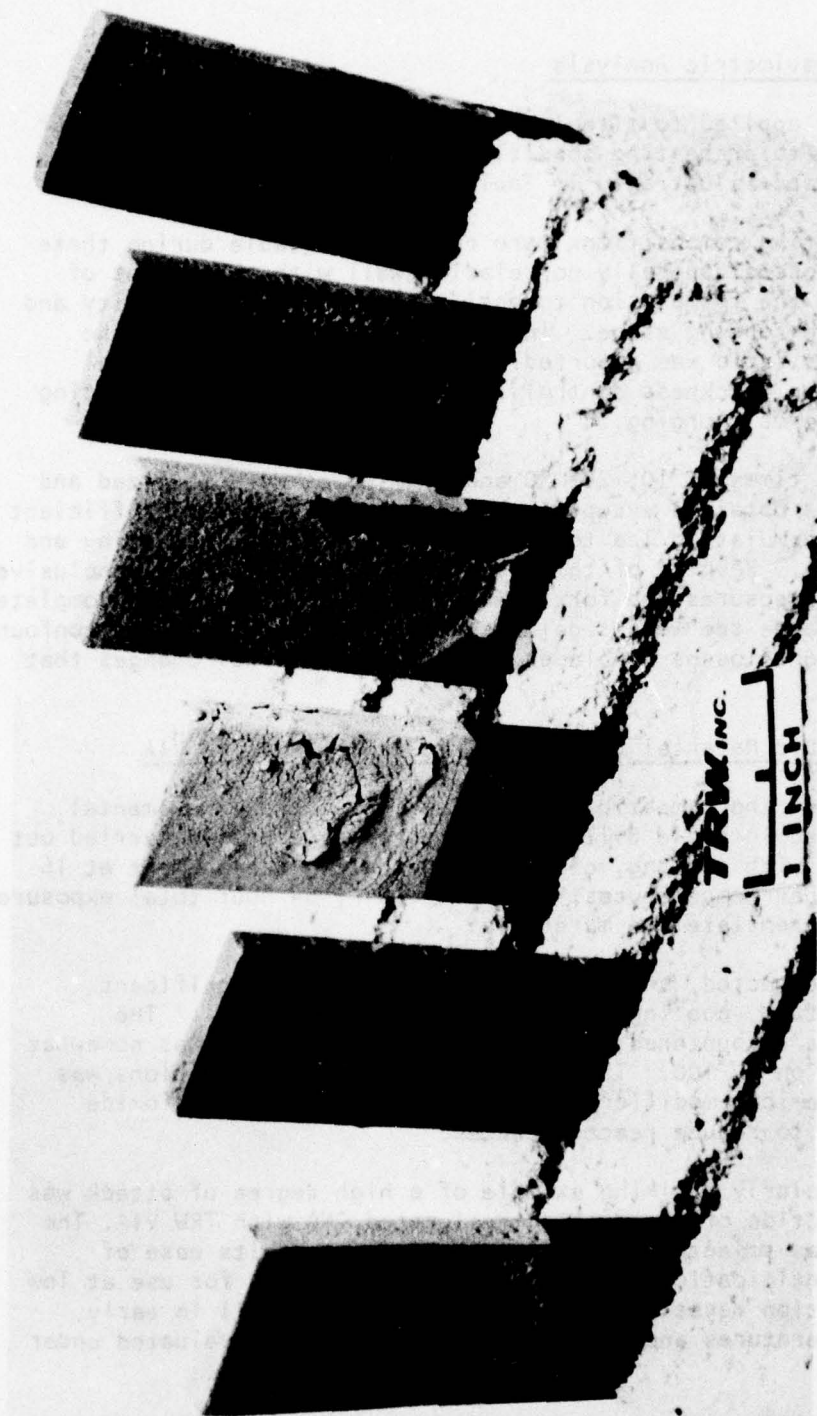
### A. Workpiece Coatings - Nickel Alloy Dies

The conventional, and almost certainly the most economical method for applying interfacial separation-lubrication materials for use in isothermal forging is as a workpiece precoat. The high affinity of titanium alloys makes a protective precoat applied prior to elevated temperature exposure virtually mandatory. It is desirable in isothermal forging to have such a protective coating formulated to also provide effective separation and lubrication without the need for die coatings. For this reason a portion of the experimental efforts were devoted to simple workpiece coating systems.

#### 1. Coating Application and Fusion

In view of the large number of possible combinations of matrix and particulate phases evident in Table I, some preliminary screening was performed by formulating trial coatings covering a spectrum of volume fraction levels with many of the particulate-matrix combinations. These formulations were applied to titanium alloy coupons and exposed in air at 1500°F or 1600°F to determine the coating's application behavior. The coatings were rated visually for appearance and the more promising candidates were subjected to X-ray diffraction analysis of the coating constituency. Figure 7 illustrates a series of samples exposed at 1600°F. No coating shown in this figure appeared promising at the relatively high particulate concentration. X-ray diffraction examination of the coatings demonstrated that the bentonite and mica formulations reacted vigorously at high concentrations to release water of hydration and form a hard, non-vitreous coating. At low volume fractions, the material rapidly dissolved in the glass. For this reason, they were eliminated as candidates. A cerium oxide-graphite combination behaved similarly at high particulate content. It had been chosen on the basis of prior work that had shown that graphite performs well as a high temperature lubricating substance in the presence of  $\text{CeO}_2$ .

The other candidate materials performed well at concentrations near 10% by weight. For further evaluation, levels of 4 and 8 percent were selected.



Cerium Oxide  
 Cerium Oxide + Graphite  
 Bentonite  
 Mica  
 Rutile  
 Titanium Carbide

FIGURE 7. Application-Fusion Evaluation Samples after 1 Hour Exposure at 1600F. Particulate Phase at 30% level in 32B Matrix.



## 2. Thermogravimetric Analysis

Coatings applied to titanium alloy coupons were evaluated for weight loss on exposure to preheating conditions at both 1300°F and 1500°F. Results of these tests are illustrated in Table B-1.

Most coating compositions were reasonably stable during these exposures with weight losses generally correlating well with the amount of organic binder added to the formulation to assist with coating continuity and handleability in the pre-forging stage. In some cases measurement of the green-to-fused thickness ratio was reported. This quantity is a critical one for assuring adequate thickness control in both ring compression testing and in production isothermal forging.

Exposure times of 10, 20, 30 and 60 minutes were utilized and good reproducibility was obtained except in a few instances where insufficient organic binder in the formulation led to discontinuous coatings, spalling and titanium alloy oxidation. Results of this type of evaluation were inconclusive for higher temperature exposures and for formulations that did not fuse completely at 1300°F or 1500°F because the weight gain caused by oxygen pickup was confounded with the effects of weight losses by binder removal and chemical changes that occurred.

## 3. Coating-Die Material Compatibility, IN-100 and TRW VIA

Results of the compatibility evaluation on the experimental formulations are reported in Table B-11. Exposures were generally carried out for a total of 80 hours with cooling, cleaning and coating replacement at 16 hour intervals. At higher temperatures, such as 1800°F, 64 hour total exposures were sufficient to differentiate the materials.

As expected, the compatibility data show significant variations with temperature, coating composition and die material. The degree of weight changes encountered increased with temperature, was somewhat greater on TRW VIA than on IN-100. The effect of particulate additions was significant, and the chemical modifiers cerium oxide and titanium dioxide showed a clear tendency to reduce reaction rates.

A particularly striking example of a high degree of attack was demonstrated by the reaction of the coating designated GNA with TRW VIA. The soda containing glass was selected for evaluation because of its ease of producibility and the anticipation that it might be acceptable for use at low temperatures where reaction rates are low. It did not fuse well in early evaluations at low temperatures and therefore was not further evaluated under those conditions.

Compatibility exposures with GNA in contact with both TRW VIA and IN-100 were made to verify these results. Some exposed samples are illustrated in Figure 8. On a duplicate sample the results were the same and visually apparent. It is clear that the sodium content of the matrix phase must be kept low to insure freedom from this attack.



**TRW INC.**



GCO  
+  
8CeO

GCO  
+  
8TiC

GFE

GNA

Control

**a. Original 80 Hour Exposure Test**



**b. Duplicate GNA Test**

**FIGURE 8: Compatibility Samples after 80 Hour Exposure on TRW VI A at 1800°F**



#### 4. Adhesion and Accumulation

Table B-III summarizes the results of the shear adhesion evaluation performed by the procedures outlined in Section II. The accumulation value reported for these tests designates the "steady-state" film thickness produced on the die material sample following a series of three simulated forgings.

The results of this evaluation demonstrate considerable variation in both shear adhesion and accumulation between formulations with the matrix phase exerting the predominant influence. Particulate type was a secondary but still significant factor.

The applied load column in this table indicates the load that was barely sufficient to cause a very slight plastic deformation on the titanium alloy sample during the two-minute load application portion of the test. As seen from the data these load requirements varied substantially with temperature for the titanium alloys.

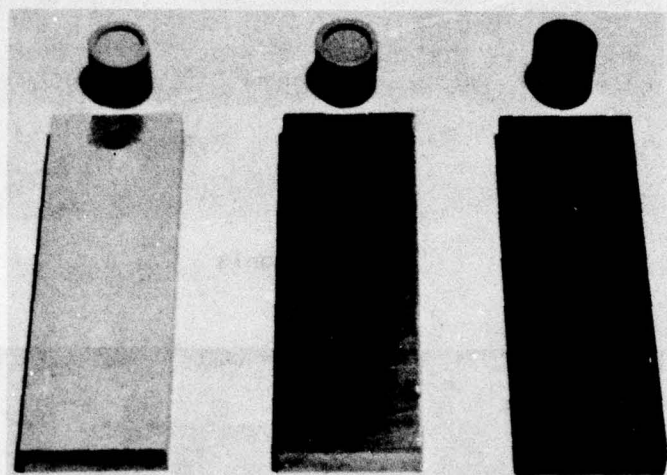
A significant amount of scatter about the reported average was encountered in the shear stress measurements. However, this was substantially lower than that reported in Section IV and the standard deviations encountered "within formulations" were small enough compared to the "between formulation" variability to allow conclusive comparisons to be made.

#### B. Die Applied Coatings

Table I lists the three die applied coating systems utilized in this program. The three tables in Appendix B present the data obtained in compatibility, adhesion and accumulation measurements with these. The coated samples are illustrated in Figure 9, while microstructural sections through control samples coated at the same time are shown in Figure 10.

It is apparent from these figures that the zirconium oxide and Ni-Al materials applied well. Coatings were uniform and close to the target thickness of 0.025 inches. The siliconized coating was non-uniform and discontinuous. The reaction mechanism responsible for the coating formation apparently was ineffective for the nickel alloy.

For the zirconia coated samples, bonding between coating and substrate shown by the micrograph of Figure 10 appeared to be simply mechanical at best. However, the as-coated samples showed excellent adhesion despite some handling in mechanically polishing the coating surface prior to testing. Bonding of the NiAl was much better, apparently metallurgical in character and uniform over the sample surface.



Zirconia

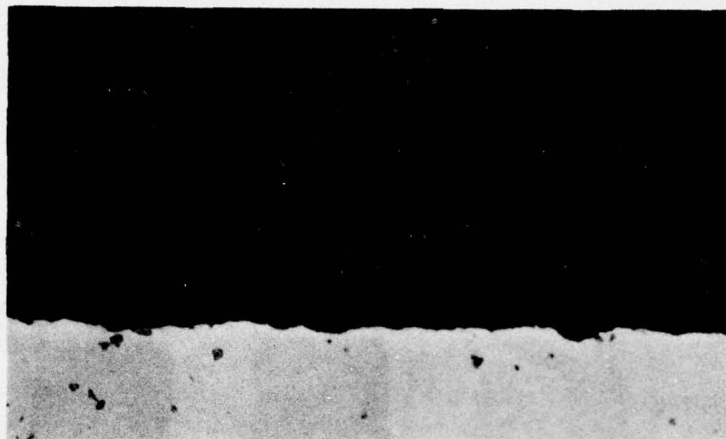
NiAl

Silicon

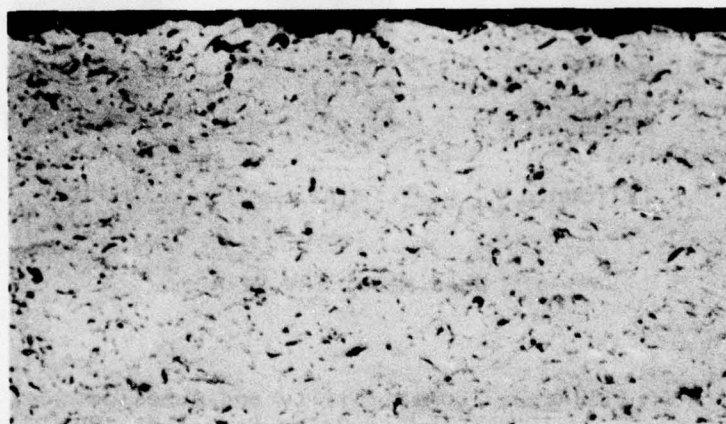
Base Metal TRW VI A

FIGURE 9. Compatibility and Adhesion  
Samples Prior to use at 1800F

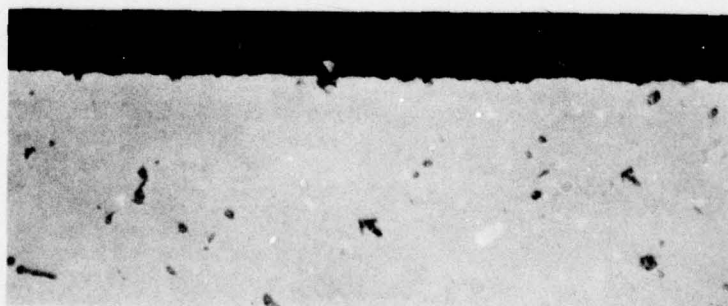




a. Zirconia



b. NiAl



c. Silicon

FIGURE 10. Microstructures of Die Applied Coatings

Evaluation results for these coatings are reported in the tables in Appendix B. Compatibility was measured in a somewhat different fashion for the coated samples. It was not practical to remove the lubricants by grit blasting since this also removed the die applied coatings. Therefore, separation-lubrication material was added to the coated samples and evaluation was performed primarily visually rather than with weight change. Nevertheless, the coatings offered some protection as evidenced by the absence of gross attack by the GNA coating such as shown in Figure 8. The exposed coated samples are shown in Figure 11 after 80 hours accumulated exposure. Some evidence of gross attack is apparent on the siliconized and pre-oxidized samples, and spalling of the zirconia coating is apparent.

The adhesion and accumulation results were more definitive. The zirconia coated surface successfully survived three adhesion cycles. On cooling to room temperature however, the coating fractured about midway through its thickness leaving approximately a 10 mil thick coating intact with the TRW VIA piece. This prevented measurement of accumulation, but it was apparent that some penetration of the lubricant into residual porosity in the film had occurred.

The siliconized surface demonstrated an adhesive character similar to the uncoated TRW VIA. No particular difficulties were encountered and yet no definite advantages were seen.

The NiAl coating however showed a very interesting effect in that with three adhesion tests run at 1800°F, no adhesion or accumulation was detected. A second set of adhesion cycles was made with a different lubricant system, and this time some very low forces were required, but the rate of accumulation was extremely small. In fact, the 0.3 mil total accumulation observed at the end of six exposures was essentially a greenish discolored surface and may have been an oxide film.

The reason for this behavior on the NiAl coating is not apparent, but seems advantageous to investigate the situation further through, for example, ring compression evaluation.

#### C. Silicon Nitride Die Insert Materials

Compatibility and adhesion measurements were performed on hot pressed and sintered silicon nitride samples. The test results are recorded in the compatibility and adhesion tables of Appendix B.

Compatibility samples from this material were exposed at 1800°F but removal of the residual coating material after exposure was not possible because of the damage incurred in grit blasting. Vapor blast and thermal treatment methods were attempted without success. Therefore, coating replacement was made after each exposure with no cleaning.





**FIGURE 11. Compatibility Test**  
Coupons following 80 Hour  
Exposure at 1800F. All samples  
TRW VI A Mod R Base Metal.

The exposed samples are illustrated in Figure 12. Part a of this figure shows the samples cooled to room temperature after a 16 hour treatment at 1800°F. The excellent fusion behavior and low degree of wetting exhibited by GNA is apparent. Part b of the figure shows the samples after 80 hours of exposure and cooled to room temperature. The lowest viscosity formulation, GFE, apparently penetrated into the sample either by an intergranular mechanism or through surface connected porosity. In any event the choice of the high viscosity, low wetting formulation is indicated.

The adhesion and accumulation data in Table B-III do not change this conclusion as not much variation in shear stress was seen. All samples of silicon nitride fractured during testing to some degree. The GNA sample showed massive fracture but this was caused by an inadvertent application of 2800 pound load. The other samples showed minor fracturing at their corners.





Uncoated  
Exposed

4

GCO

3

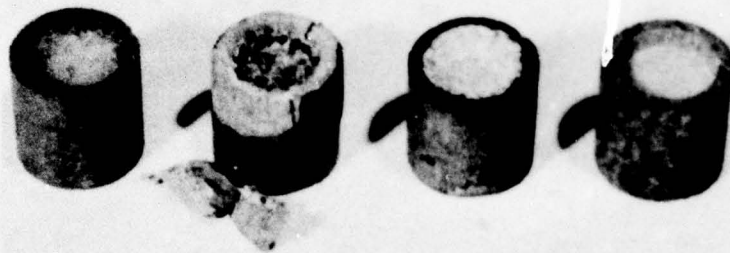
GFE

2

GNA

1

a. After 16 Hour Exposure at 1800F.



GNA

1

GFE

2

GCO

3

Uncoated  
Exposed

4

b. After 80 Hour Total Exposure at 1800F

FIGURE 12. Silicon Nitride Compatibility Samples.

## VI. SEPARATION-LUBRICATION SUBSTANCES FOR FURTHER EVALUATION

The following is a list of formulations that were selected for further evaluation by ring compression testing at the Westinghouse Astronuclear Laboratory hot die facility at Wright-Patterson Air Force Base, Ohio:

1) Code: GPC4

Service range 1300-1400°F  
on nickel alloy dies

### Formulation (parts by weight):

Matrix	22A	- 470
Particulate	Ce363	- 42
Binder	ACR-1	- 101
Diluant	ISO	- 510

2) Code: GBTC8

Service range 1300-1400°F  
on nickel alloy dies

### Formulation (parts by weight):

Matrix	32B	- 600
Particulate	Ti276	- 40
Binder	ACR	- 47
Diluant	XYL	- 70

3) Code: GPT4

Service range 1500-1600°F  
on nickel alloy dies

### Formulation (parts by weight):

Matrix	22A	- 400
Particulate	Ti279	- 24
Binder	ACR-1	- 86
Diluant	XYL	- 440



4) Code: GFBN8

Service range 1700-1800°F  
on TRW VIA dies

Formulation (parts by weight):

Matrix	GFE	- 300
Particulate	BN129	- 24
Binder	ACR-1	- 446
Diluant	XYL	- 77

5) Code: GFTC8

Service range 1700-1800°F  
on TRW VIA dies

Formulation (parts by weight):

Matrix	GFE	- 300
Particulate	Ti276	- 24
Binder	ACR-1	- 78
Diluant	XYL	- 460

6) Code: GCO

Service range 1700-1800°F  
on NiAl coated dies

Formulation (parts by weight):

Matrix	GCO	- 250
Binder	ACR-1	- 63
Diluant	XYL	- 380

7) Code: GNA

Service range 1700-1800°F  
with silicon nitride die insert

Formulation (parts by weight):

Matrix	GNA	- 600
Binder	ACR-1	- 96
Diluant	ISO	- 356

## VII. SUMMARY AND CONCLUSIONS

A variety of workpiece and die coatings were developed and formulated on this program to act as interface separation-lubrication substances at specific temperatures ranging between 1300°F and 1800°F. Many of these were evaluated for their fusion and thermogravimetric stability, chemical compatibility with die materials, adhesion properties in shear, and steady state film accumulation under conditions simulating those encountered in hot-die-isothermal or near-isothermal forging practice. Similar properties of several "state-of-technology" compositions were evaluated near a typical use temperature of 1650°F.

Comparison of the data characterizing the advanced compositions with the baseline provided by the prior formulations revealed that several new substances show superior properties and merit further examination by ring compression testing and later by prototype isothermal forging practice. Quantities of the most promising experimental separation-lubrication substances have been supplied for these evaluations.



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**SPECTROCHEMICAL ANALYSIS DATA**

Element	CAT	CATMA	WO-JIS	W-STEEL	DET JIS
Al	-	-	Min.	Min.	Min.
C	Major	Major	Min.	Min.	Major
Co	Trace	-	Min.	Min.	Min.
Cr	-	-	Min.	Min.	-
Fe	Trace	Trace	Min.	Min.	Trace
Mo	Trace	Trace	Trace	Trace	Trace
Ni	-	-	-	-	-
P	-	-	-	-	-
S	-	-	Min.	Min.	-
Si	Min.	Major	Min.	Min.	Major
Ti	-	-	-	-	-
V	-	-	Min.	Min.	-
W	-	-	Min.	Min.	-
Zn	Trace	Trace	-	-	Trace
Cu	Min.	Min.	-	-	Major
Ba	-	-	-	-	-
Li	-	-	-	-	-
Mn	-	-	-	-	-
Na	-	-	-	-	-
Pb	-	-	-	-	-
Sn	Trace	Trace	-	-	Trace
Te	-	-	-	-	-
Zr	-	-	-	-	-

**APPENDIX A**

**EVALUATION DATA FOR  
STATE OF TECHNOLOGY ASSESSMENT**

Major - Major  
Minor - Minor  
Trace

Major analysis performed by oxygen combustion.

TABLE A-1  
SPECTROCHEMICAL ANALYSIS DATA

<u>Element</u>	<u>Lubricant Formulation</u>				
	<u>CRT</u>	<u>CRT-HA</u>	<u>WO-315</u>	<u>D-347M</u>	<u>OPT 112</u>
Al	-	-	Min.	Min.	Min.
B	Maj.	Maj.	Min.	Min.	Maj.
Ca	Trace	-	Min.	Min.	Min.
Cr	-	-	Min.	Min.	-
Fe	Trace	Trace	Min.	Min.	Trace
Mg	Trace	-	Trace	Trace	Trace
Mn	-	-	-	-	-
Pb	-	-	-	-	-
Si	Min.	Maj.	Min.	Min.	Maj.
Ti	-	-	-	-	-
K	-	-	Min.	Min.	-
Na	-	-	Min.	Min.	-
Ni	Trace	Trace	-	-	Trace
Co	Min.	Min.	-	-	Maj.
Ba	-	-	-	-	-
Li	-	-	-	-	-
Zn	-	-	-	-	-
Zr	-	-	-	-	-
Sb	-	-	-	-	-
Cu	Trace	Trace	-	-	Trace
Sr	-	-	-	-	Trace
S*	.005	.003	.009	.008	.015

Maj. → Major  
Min. → Minor  
Trace

\*Sulfur analysis performed by oxygen combustion.



TABLE A-11  
VISUAL STABILITY TESTS - COATING THICKNESS DATA

<u>Lubricant</u>	<u>Measurement Time</u>	<u>Rod Diam</u>	<u>Station</u>				
			<u>0.500</u>	<u>0.700</u>	<u>0.900</u>	<u>1.100</u>	<u>1.300</u>
CRT	Before	0.2576	0.2696	0.2685	0.2698	0.2690	0.2698
	After	-	0.2606	0.2613	0.2604	0.2613	0.2609
OPT 112	Before	0.2555	0.2649	0.2640	0.2640	0.2641	0.2639
	After	-	0.2592	0.2600	0.2602	0.2608	0.2600
CRT-HA	Before	0.2571	0.2698	0.2698	0.2694	0.2680	0.2692
	After	-	0.2691	0.2644	0.2685	0.2640	0.2669
D 347M	Before	0.2573	0.2671	0.2677	0.2677	0.2668	0.2681
	After	-	0.2608	0.2591	0.2599	0.2561	0.2598
WO 315	Before	0.2535	0.2683	0.2678	0.2656	0.2678	0.2666
	After	-	0.2603	0.2611	0.2610	0.2610	0.2617

- All samples exposed 3 Hours in air at 1750°F
- Thicknesses (in inches) measured on 10X optical comparator
- "Station" refers to distance from edge of coating

TABLE A-III

LUBRICANT THERMOGRAVIMETRIC ANALYSIS

Coating	Exposure		Before Exposure		After Exposure		Coating Thickness (Mils/Side)
	Time (Min)	Temp (°F)	Coating Weight (Grams)	Coating Thickness (Mils/Side)	Coating Weight (Grams)	Weight Change (%)	
D-347M	60	600	0.1320	0.0026	0.0097	25.2	0.0022
"	10	1300	0.1533	0.0028	0.1067	30.4	0.0022
"	20	1300	0.1403	0.0027	0.0982	30.0	0.0020
"	30	1300	0.1420	0.0026	0.0992	30.1	0.0018
"	60	1300	0.1428	0.0026	0.1005	29.6	0.0018
"	180	1300	0.1364	0.0025	0.0978	28.3	0.0015
"	240	1300	0.1415	0.0026	0.0999	29.3	0.0018
"	10	1700	0.1446	0.0026	0.0985	31.9	0.0014
"	20	1700	0.1457	0.0026	0.1002	31.2	0.0009
"	30	1700	0.1428	0.0026	0.0970	32.0	0.0008



TABLE A-111 (CONTINUED)

LUBRICANT THERMOGRAVIMETRIC ANALYSIS

Coating	Exposure		Before Exposure		After Exposure		Coating Thickness (Mils/Side)
	Time (Min)	Temp (°F)	Coating Weight (Grams)	Coating Thickness (Mils/Side)	Coating Weight (Grams)	Weight Change (%)	
CRT	60	600	0.1722	0.0032	0.1350	21.6	0.0026
"	10	1300	0.1989	0.0034	0.1473	26.0	0.0029
"	20	1300	0.1817	0.0034	0.1355	25.4	0.0014
"	30	1300	0.1885	0.0034	0.1388	26.4	0.0014
"	60	1300	0.2008	0.0037	0.1511	24.8	0.0014
"	180	1300	0.2310	0.0042	0.1691	26.8	0.0017
"	240	1300	0.2231	0.0044	0.1674	25.0	0.0016
"	10	1700	0.1854	0.0035	0.1402	24.3	0.0012
"	20	1700	0.2092	0.0033	0.1607	23.1	0.0014
"	30	1700	0.1871	0.0032	0.1431	23.5	0.0012

TABLE A-III (CONTINUED)

## LUBRICANT THERMOGRAVIMETRIC ANALYSIS

Coating	Exposure		Before Exposure		After Exposure		Coating Thickness (Mils/Side)
	Time (Min)	Temp (°F)	Coating Weight (Grams)	Coating Thickness (Mils/Side)	Coating Weight (Grams)	Weight Change (%)	
CRT-HA	60	600	0.1424	0.0027	0.1191	16.4	0.0024
"	10	1300	0.1633	0.0028	0.1332	18.4	0.0012
"	20	1300	0.1735	0.0030	0.1419	18.2	0.0012
"	30	1300	0.1686	0.0030	0.1377	18.3	0.0012
"	60	1300	0.1683	0.0031	0.1381	18.0	0.0010
"	180	1300	0.1692	0.0028	0.1393	17.7	0.0010
"	240	1300	0.1621	0.0028	0.1310	19.1	0.0010
"	10	1700	0.1565	0.0028	0.1284	18.0	0.0010
"	20	1700	0.1614	0.0028	0.1342	16.9	0.0012
"	30	1700	0.1669	0.0025	0.1382	17.1	0.0010



TABLE A-111 (CONTINUED)  
LUBRICANT THERMOGRAVIMETRIC ANALYSIS

Coating	Exposure		Before Exposure		After Exposure		Coating Thickness (Mils/Side)
	Time (Min)	Temp (°F)	Coating Weight (Grams)	Coating Thickness (Mils/Side)	Coating Weight (Grams)	Weight Change (%)	
W0 315	60	600	0.1750	0.0030	0.1579	9.8	0.0030
"	10	1300	0.1849	0.0037	0.1560	15.6	0.0030
"	20	1300	0.2089	0.0046	0.1770	15.3	0.0044
"	30	1300	0.2312	0.0046	0.1964	15.0	0.0048
"	60	1300	0.2169	0.0046	0.1842	15.0	0.0048
"	180	1300	0.2312	0.0048	0.1979	14.4	0.0050
"	240	1300	0.2434	0.0053	0.2056	15.5	0.0058
"	10	1700	0.3011	0.0050	0.2480	17.6	0.0193
"	20	1700	0.2082	0.0032	0.1740	17.7	0.0068
"	30	1700	0.2239	0.0046	0.1858	17.0	0.0055
OPT-112	60	600	0.1677	0.0028	0.1139	32.0	0.0024
"	10	1300	0.1087	0.0020	0.0752	30.8	0.0013
"	20	1300	0.0972	0.0020	0.0639	34.2	0.0010
"	30	1300	0.1024	0.0022	0.0690	32.6	0.0014
"	60	1300	0.1022	0.0023	0.0693	32.2	0.0012
"	180	1300	0.0968	0.0025	0.0657	32.1	0.0010
"	240	1300	0.1012	0.0022	0.0677	33.1	0.0010
"	10	1700	0.0969	0.0024	0.0663	31.6	0.0012
"	20	1700	0.0927	0.0020	0.0638	31.1	0.0010
"	30	1700	0.0975	0.0022	0.0675	29.0	0.0012

TABLE A-IV  
COATING - IN-100 COMPATIBILITY

Exposure Time (Hours)	CG 44	CRT	CRT-HA	D-347M	W0-315	OPT 112	Uncoated Exposed Sandblasted	Uncoated Unexposed Sandblasted
16	0.21	0.49	0.26	0.15	0.38	0.17	0.08	0.14
32	0.45	1.04	0.71	0.42	0.79	0.46	0.32	0.35
48	0.82	1.70	1.63	0.82	1.34	1.04	0.71	0.61
64	1.45	2.33	2.43	1.42	1.93	1.82	0.99	0.82
80	1.94	2.96	3.28	1.83	2.53	2.16	1.31	1.08
96	2.21	3.47	3.76	2.17	2.95	2.46	1.53	1.30
112	2.95	5.06	5.22	2.85	3.86	3.29	1.99	1.49
128	3.41	5.61	5.81	3.19	4.38	3.74	2.21	1.75
144	3.69	6.06	6.25	3.40	4.67	4.03	2.37	1.88
160	3.89	6.58	6.57	3.61	5.03	4.42	2.54	2.06
176	4.02	6.88	6.91	3.85	5.40	4.67	2.71	2.27
192	4.16	6.96	7.09	4.05	5.47	4.89	-	-
208	4.40	7.09	7.26	4.22	5.71	5.19	-	-
Appearance	Thick Matte Non-wetting(?)	Glossy Smooth	Glossy Smooth	Blistered	Slightly Blistered	Smooth Matte		
Color as Fused	White	Violet	Deep Violet	Green	Dark Green	Light Gray		

Table entries are cumulative weight loss.  
For IN-100 sample, expressed as percent of original weight.  
Amount of lubricant applied (solids only) 102±5 milligrams.



TABLE A-V

## COATING ADHESION MEASUREMENTS

Test No.	Lubri-cant	Dried Coating Thickness (Mils)	Load (Lbs)	Shear Stress (PSI)	Shear Creep (In.)	Material		Heat Time (Min.)	Comment
1	CRT	12	2030	5.6	-	Ti*	Ti*	10	Uncertainty in strain gage readings because of heat buildup
2	"	"	1950	17.0	-	"	"	"	
3	"	"	1780	34.6	-	"	"	"	
4	"	"	1880	28.7	-	"	"	"	
5	"	"	1870	26.8	-	"	"	"	
6	"	"	1800	39.2	-	"	"	"	
7	"	"	2950	26.8	-	"	"	"	
8	"	"	2900	23.4	.096	"	"	"	
9	"	"	2860	20.0	.090	"	"	"	
10	"	6	2804	16.7	.130	"	"	"	
46	"	6	3100	22.2	0	Ti*	IN 100	20	
47	"	6	2980	35.0	0	Ti*	IN 100	"	
48	"	6	2940	16.2	0	Ti*	IN 100	"	
17	"	6	3150	55.0	.163	Ti*	Ti	"	
67	"	6	2910	31.7	0	Ti*	IN 100	"	
68	"	6	3070	25.8	0	Ti*	IN 100	"	
69	"	6	3030	35.6	0	Ti*	IN 100	"	
11	CRT-HA	12	2890	38.8	.173	Ti*	Ti*	10	For all tests - Exposure Temperature - 1650°F Dwell Under Load - 2 min
12	"	12	2930	30.5	.154	Ti*	Ti*	"	
18	"	6	2910	33.0	.077	Ti*	Ti	"	

\*Asterisks designate coated samples.

For all tests -

Exposure Temperature - 1650°F  
Dwell Under Load - 2 min

TABLE A-V (CONTINUED)

## COATING ADHESION MEASUREMENTS

Test No.	Lubri-cant	Dried Coating Thickness (Mils)	Load (Lbs)	Shear Stress (PSI)	Shear Creep (in.)	Material		Heat Time (Min.)	Comment
29	W0 315	12	2970	71.8	0	Ti*	Ti*	10	
30	"	"	3010	67.4	0	"	"	"	
31	"	"	3190	59.9	0	"	"	"	
38	"	6	3000	50.8 (Max)	0	"	Ti	20	Insufficient air pressure
39	"	"	2990	16.4	0	"	IN 100	"	
43	"	"	2840	21.9	.127	"	"	"	
44	"	"	2900	24.5	.178	"	"	"	
45	"	"	3030	26.1	.127	"	"	"	
55	"	"	2910	37.5	.102	"	"	"	
56	"	"	2930	34.2	.089	"	"	"	
57	"	"	2830	30.8	.083	"	"	"	
14	OPT 112	8	2920	22.0	.192	Ti*	Ti*	10	
19	"	4	3080	18.7	.067	Ti	Ti	"	
32	"	12	3030	14.7	.191	Ti*	Ti*	"	
33	"	"	3000	26.1	.064	"	"	"	
34	"	"	2950	27.0	0	"	"	"	
35	"	"	3000	29.4	0	"	"	30	
36	"	6	3050	46.6	.070	"	"	20	
37	"	"	3050	13.3	0	Ti	IN 100	"	
40	"	"	-	11.3	0	"	"	"	
41	"	"	3000	10.5	0	"	"	"	Recorder malfunction on load
42	"	"	3100	11.1	0	"	"	"	
61	"	"	2840	14.8	0	"	"	"	
62	"	"	3000	15.5	0	"	"	"	
63	"	"	3070	13.1	0	"	"	"	
16	CG 44	12	3170	5.7	0	Ti*	Ti*	10	
20	"	6	3000	20.0	.096	"	Ti	"	

\*Asterisks designate coated samples.



TABLE - V (CONTINUED)

## COATING ADHESION MEASUREMENTS

Test No.	Lubri-cant	Dried Coating Thickness (Mils)	Load (Lbs)	Shear Stress (PSI)	Shear Creep (In.)	Material		Heat Time (Min.)	Comment
						Upper Plate	Lower Plate		
49	CRT-HA	6	2990	33.0	0	Ti*	IN 100	20	
50	"	"	2880	20.2	0	Ti*	IN 100	"	
51	"	12	2930	32.5	0	Ti*	Ti*	"	
52	"	6	2960	48.2	0	Ti*	IN 100	"	
53	"	"	2970	34.1	0	Ti*	IN 100	"	
54	"	"	2970	38.8	0	Ti*	IN 100	"	
64	"	6	2990	-	0	Ti*	Ti*	"	- Recorder malfunction
65	"	6	3100	25.6	0	Ti*	Ti*	"	
66	"	6	3040	25.3	0	Ti*	Ti*	"	
13	347M	12	2920	30.0	.144	Ti*	Ti*	10	
22	"	"	3010	30.6	1.0	Ti*	Ti*	"	
26	"	6	2920	59.2	.173	Ti*	Ti	"	
27	"	"	2970	62.2	.672	Ti*	Ti	"	
28	"	"	2950	40.7	.154	Ti*	Ti	"	
58	"	"	2930	34.9	.114	Ti*	IN 100	20	
59	"	"	2930	48.2	.102	Ti*	IN 100	"	
60	"	"	2910	39.5	.089	Ti*	IN 100	"	
15	W0 315	10	2960	20.5	.182	Ti*	Ti*	10	
21	"	6	3040	62.2	.154	"	"	"	
23	"	"	2920	39.8	.202	Ti*	Ti	"	Samples overlapped more than 1" because of oxidized pins. Rear pull rod replaced with refractory alloy.
24	"	"	2910	147.0	0	"	"	"	
25	"	"	2970	61.0	.250	"	"	"	

\*Asterisks designate coated samples.

## APPENDIX B

### EVALUATION DATA FOR ADVANCED FORMULATIONS



TABLE B-1  
LUBRICANT THERMOGRAVIMETRIC ANALYSIS

Coating	1300°F Exposure Time (Min)	Before Exposure		After Exposure		Weight Loss (%)	Weight Loss (mg/cm <sup>2</sup> )	Thickness Ratio Fused/Green (%)
		Coating Weight (Grams)	Coating Thickness (Mils/Side)	Coating Weight Loss (Grams)	Coating Thickness (Mils/Side)			
44A	10	0.0525	NR	0.0080	NR	15.2	0.6	NR
	20	0.0687	"	0.0092	"	13.4	0.7	"
	30	0.0538	"	0.0078	"	14.5	0.6	"
	60	0.0672	"	0.0091	"	13.5	0.7	"
44A+8% CeO	10	0.2840	"	0.0270	"	9.5	2.0	"
	20	0.2616	"	0.0262	"	10.0	2.0	"
	30	0.2521	"	0.0270	"	10.7	2.0	"
	60	0.2575	"	0.0280	"	10.9	2.1	"
44A+8% TiC	10	0.1580	"	0.0195	"	12.3	1.5	"
	20	0.1145	"	0.0186	"	16.2	1.4	"
	30	0.1000	"	0.0177	"	17.7	1.3	"
	60	0.0993	"	0.0185	"	18.6	1.4	"
22A	10	0.1153	"	0.0019	"	1.7	0.1	"
	20	0.1121	"	0.0009	"	0.8	0.1	"
	30	0.1046	"	0.0013	"	1.2	0.1	"
	60	0.1007	"	0.0012	"	1.2	0.1	"
22A+8% TiO <sub>2</sub>	10	0.0904	"	0.0042	"	4.7	0.3	*
	20	0.0851	"	0.0042	"	4.9	0.3	"
	30	0.0846	"	0.0036	"	4.3	0.3	"
	60	0.0951	"	0.0037	"	3.9	0.3	"

\*Coatings spalled - apparently insufficient binder

TABLE B-1 (CONTINUED)  
LUBRICANT THERMOGRAVIMETRIC ANALYSIS

Coating	Exposure 1300°F Time (Min)	Before Exposure		After Exposure		Weight Loss (%)	Weight Loss (mg/cm <sup>2</sup> )	Thickness Ratio Fused/Green (%)
		Coating Weight (Grams)	Coating Thickness (Mils/Side)	Coating Weight Loss (Grams)	Coating Thickness (Mils/Side)			
22A+8% TiC	10	0.0470	NR	0.0011	NR	2.3		NR
	20	0.0481	"	-	"	-		"
	30	0.0459	"	-0.0004	"	-0.9		"
	60	0.0503	"	-0.0010	"	-2.0		"
32B+8% TiC	10	0.1155	0.0043	0.0292	0.0016	25.3	2.2	37
	20	0.1237	0.0053	0.0304	0.0016	24.6	2.8	30
	30	0.1275	0.0049	0.0308	0.0012	24.1	2.3	25
	60	0.1325	0.0051	0.0324	0.0015	24.4	2.4	29
32B+8% TiO <sub>2</sub>	10	0.1155	0.0043	0.0292	0.0016	25.3	2.2	37
	20	0.1237	0.0053	0.0304	0.0016	24.6	2.8	30
	30	0.1275	0.0049	0.0308	0.0012	24.1	2.3	25
	60	0.1325	0.0051	0.0324	0.0015	24.4	2.4	29
32B+8% TiO <sub>2</sub>	10	0.0984	0.0046	0.0252	0.0037	25.5	1.9	80
	20	0.1002	0.0051	0.0250	0.0038	25.0	1.9	75
	30	0.0982	0.0051	0.0246	0.0045	25.1	1.8	88
	60	0.1005	0.0050	0.0259	0.0020	25.8	1.9	60
22A	10	0.2103	0.0030	0.0181	0.0018	8.6	1.4	60
	20	0.2349	0.0036	0.0185	0.0022	7.9	1.4	60
	30	0.2074	0.0030	0.0185	0.0015	8.9	1.4	50
	60	0.2384	0.0037	0.0200	0.0016	8.4	1.5	43

\*Coatings spalled - apparently insufficient binder



TABLE B-1 (CONTINUED)  
LUBRICANT THERMOGRAVIMETRIC ANALYSIS

Coating	1300°F Exposure Time (Min)	Before Exposure		After Exposure		Weight Loss (%)	Weight Loss (mg/cm <sup>2</sup> )	Thickness Ratio Fused/Green (%)
		Coating Weight (Grams)	Coating Thickness (Mils/Side)	Coating Weight Loss (Grams)	Coating Thickness (Mils/Side)			
22A+8% TiO <sub>2</sub>	10	0.7114	0.0123	0.0437	0.0061	6.1	3.3	50
	20	0.7178	0.0133	0.0427	0.0066	6.0	3.2	50
	30	0.1750	0.0038	0.0106	0.0027	6.1	0.8	71
	60	0.1607	0.0048	0.0095	0.0038	5.9	0.7	79
22A+8% TiC	10	0.0948	0.0022	0.0090	0.0024	9.5	0.7	109
	20	0.0836	0.0023	0.0052	0.0018	6.2	0.4	78
	30	0.0932	0.0021	0.0058	0.0017	6.2	0.4	80
	60	0.0886	0.0023	0.0045	0.0016	5.1	0.3	70
38BB	10	0.0813	0.0021	0.0078	0.0015	9.6	0.6	71
	20	0.0840	0.0020	0.0075	0.0013	8.9	0.6	62
	30	0.0831	0.0022	0.0075	0.0012	9.0	0.6	52
	60	0.0812	0.0021	0.0073	0.0011	9.0	0.6	48
38BB+8% TiC	10	0.0863	0.0023	0.0073	0.0016	8.5	0.6	69
	20	0.0856	0.0024	0.0063	0.0017	7.4	0.5	73
	30	0.0872	0.0023	0.0065	0.0016	7.5	0.5	71
	60	0.0866	0.0022	0.0072	0.0017	8.3	0.5	76
38BB+8% CeO	10	0.2201	0.0041	0.0101	0.0059	4.6	0.8	143
	20	0.1704	0.0044	0.0100	0.0046	5.8	0.8	105
	30	0.0740	0.0045	0.0096	0.0046	13.0	0.7	102
	60	0.0286	0.0031	0.0104	0.0035	36.4	0.8	112

TABLE B-1 (CONTINUED)  
LUBRICANT THERMOGRAVIMETRIC ANALYSIS

1350°F Exposure		Before Exposure		After Exposure		Weight Loss (%)	Weight Loss (mg/cm <sup>2</sup> )	Thickness Ratio Fused/Green (%)
Coating	Time (Min)	Coating Weight (Grams)	Coating Thickness (Mils/Side)	Coating Weight Loss (Grams)	Coating Thickness (Mils/Side)			
32B	10	0.2566	0.0069	0.2040	0.0077	20.5	3.9	115
	20	0.2394	0.0072	0.1897	0.0054	20.8	3.7	75
32B (Diluted)	10	0.2116	0.0075	0.1607	0.0042	24.1	3.8	56
	20	0.1989	0.0077	0.1527	0.0029	23.2	3.5	37



TABLE B-1 (CONTINUED)  
LUBRICANT THERMOGRAVIMETRIC ANALYSIS

Coating	1500°F Exposure Time (Min)	Before Exposure		After Exposure		Weight Loss (%)	Weight Loss (mg/cm <sup>2</sup> )	Thickness Ratio Fused/Green (%)
		Coating Weight (Grams)	Coating Thickness (Mils/Side)	Coating Weight Loss (Grams)	Coating Thickness (Mils/Side)			
32B	10	0.2345	NR	0.0699	NR	29.8	5.2	NR
	20	0.2540	"	0.0799	"	31.5	6.0	"
	30	0.2472	"	0.0824	"	33.3	6.2	"
	60	0.2420	"	0.0814	"	33.6	6.1	"
44A+8% CeO	10	0.1336	"	0.0043	"	3.2	0.3	"
	20	0.1098	"	0.0024	"	2.2	0.2	"
	30	0.1085	"	0.0013	"	1.2	0.1	"
	60	0.1175	"	0.0006	"	0.5	0.0	"
44A+8% TiO <sub>2</sub>	10	0.3893	"	0.0315	"	8.1	2.4	"
	20	0.3072	"	0.0292	"	9.5	2.2	"
	30	0.3524	"	0.0307	"	8.7	2.2	"
	60	0.2917	"	0.0266	"	9.1	2.0	"
32B+8% BN	10	0.0425	"	0.0090	"	21.2	0.7	"
	20	0.0507	"	0.0106	"	20.9	0.8	"
	30	0.0541	"	0.0114	"	21.1	0.9	"
	60	0.0501	"	0.0102	"	20.4	0.8	"
388B+8% CeO	10	0.1347	0.0051	-	0.0012	-	-	24
	20	0.1369	0.0041	0.0700	0.0023	51%	5.3	178
	30	0.1304	0.0044	0.1717	0.0018	132%	12.8	244
	60	0.1425	0.0046	0.1729	0.0022	121%	13.0	209

TABLE B-1 (CONTINUED)  
LUBRICANT THERMOGRAVIMETRIC ANALYSIS

Coating	1500°F Exposure Time (Min)	Before Exposure		After Exposure		Weight Loss (%)	Weight Loss (mg/cm <sup>2</sup> )	Thickness Ratio Fused/Green (%)
		Coating Weight (Grams)	Coating Thickness (Mils/Side)	Coating Weight Loss (Grams)	Coating Thickness (Mils/Side)			
22A	10	0.1971	0.0031	0.0158	0.0013	8.0	1.2	42
	20	0.2580	0.0042	0.0186	0.0022	7.2	1.4	52
	30	0.1967	0.0034	0.0148	0.0015	7.5	1.1	44
	60	0.2098	0.0035	0.0140	0.0016	6.7	1.1	46
22A+8% TIC	10	0.0959	0.0026	-	-	-	-	-
	20	0.0963	0.0024	-0.0539	0.0039	-55.0	-4.0	164
	30	0.0926	0.0024	-0.1562	0.0037	-169.0	-11.7	155
	60	0.0941	0.0022	-0.1583	0.0035	-168.0	-11.9	157
32B	10	0.2655	0.0070	0.0856	0.0049	32.2	6.4	69
	20	0.2739	0.0077	0.0864	0.0049	31.5	6.5	64
	30	0.2810	0.0082	0.0882	0.0047	31.4	6.6	57
	60	0.2784	0.0076	0.0884	0.0041	31.8	6.6	54
38BB	10	0.0783	0.0021	0.0064	0.0011	8.2	0.5	50
	20	0.0700	0.0020	0.0056	0.0009	8.0	0.4	46
	30	0.0745	0.0020	0.0058	0.0010	7.8	0.4	51
	60	0.0714	0.0018	0.0053	0.0012	7.4	0.4	57
32B+8% BN	10	0.1141	0.0072	0.0303	0.0046	26.6	2.3	64
	20	0.1074	0.0051	0.0296	0.0042	27.6	2.2	58
	30	0.1082	0.0057	0.0293	0.0047	27.1	2.2	82
	60	0.1091	0.0056	0.0297	0.0052	27.2	2.2	93



TABLE B-1 (CONTINUED)  
LUBRICANT THERMOGRAVIMETRIC ANALYSIS

Coating	1500°F Exposure		Before Exposure		After Exposure		Weight Loss (%)	Weight Loss <sub>2</sub> (mg/cm <sup>2</sup> )	Thickness Ratio Fused/Green (%)
	Time (Min)	Coating Weight (Grams)	Coating Thickness (Mils/Side)	Coating Weight Loss (Grams)	Coating Thickness (Mils/Side)				
22A+8% TiO <sub>2</sub>	10	0.7463	0.0134	0.0440	0.0074	5.9	3.3	0.55	
	20	0.7631	0.0128	0.0450	0.0075	5.9	3.5	0.58	
	30	0.7716	0.0124	0.0460	0.0058	6.0	3.5	0.47	
	60	0.7009	0.0136	0.0416	0.0055	5.9	3.1	0.40	
44A	10	0.3423	NR	0.0306	NR	8.9	2.3	NR	
	20	0.2980	"	0.0258	"	8.7	1.9	"	
	30	0.2852	"	0.0258	"	9.1	1.9	"	
	60	0.2913	"	0.0259	"	8.9	1.9	"	

TABLE B-11  
COATING VS. DIE MATERIAL COMPATIBILITY\*

Temperature (°F)	Die Material	Exposure Time (Hours)	44A	44A + 8 CeO <sub>2</sub>	44A + 8 TiC	32B	32B + 8 TiC
1300	IN 100	16	.04	.07	.06	.11	.07
		32	.22	.27	.15	.32	.29
		48	.74	.56	.55	.43	.48
		64	1.03	.87	.79	.59	.69
		80	1.18	1.05	.87	.75	.84
1300	IN 100		22A + 8 TiO <sub>2</sub>	22A + 8 TiC	22A + 4 CeO <sub>2</sub>	22A	
		16	.04	.01	.05	.06	
		32	.20	.16	.19	.19	
		48	.56	.81	.63	.80	
		64	.78	1.13	.85	1.09	
		80	.88	1.25	1.01	1.20	
1500	IN 100		44A	44A + 8 CeO <sub>2</sub>		32B	32B + 8 BN
		16	.17	.15		.12	.17
		32	.47	.30		.41	.58
		48	.93	.67		.85	1.10
		64	1.15	.76		1.06	1.48
		80	1.33	1.10		1.26	1.77

\*Table entries are cumulative weight loss expressed as percentage of original sample weight.



TABLE B-11 (CONTINUED)  
COATING VS. DIE MATERIAL COMPATIBILITY\*

Temperature (°F)	Die Material	Exposure Time (Hours)	22A	22A + 8 TiO <sub>2</sub>	22A + 4 TiC	D347M	D69	OPT 112
1500	IN 100	16	.17	.07	.10			
		32	.44	.34	.34			
		48	.93	.74	1.30			
		64	1.23	.99	1.84			
		80	1.73	1.18	2.50			
1650	IN 100	16	.07	.35	.12	.01	.24	.03
		32	.10	.69	.36	.07	.44	.11
		48	.11	.99	.92	.11	.63	.33
		64	.46	1.34	1.44	.43	.94	.83
		80	.63	1.94	1.97	.52	1.22	.85
		96	.68	3.07	2.23	.64	1.42	.93
1800	IN 100	16	.13	.14	.17	.27	.38	.20
		32	.43	.50	.52	.62	1.17	.56
		48	.89	.95	1.05	1.20	2.13	1.02
		64	1.20	1.24	1.43	1.55	2.90	1.32
1800	TRW VIA	16	.24	.20	.28	.31	.39	.25
		32	.50	.47	.55	.66	1.04	.38
		48	1.06	.94	1.21	1.28	2.86	.85
		64	1.50	1.28	1.46	1.57	4.91	1.21

\*Table entries are cumulative weight loss expressed as percentage of original sample weight.

TABLE B-11 (CONTINUED)

WORKPIECE COATING VS. COATED DIE-COMPATIBILITY\*

<u>Temperature (°F)</u>	<u>Die Material</u>	<u>Exposure Time (Hours)</u>	<u>Zirconia</u>	<u>Siliconized</u>	<u>NiAl</u>	"GNA" applied to sample surface in all tests
1800	Coated TRW VIA	16	-.03	-.04	-.14	
		32	-.01	-.01	-.02	
		48	0	0	-.01	
		64	0	0	.01	
		80	+.01	.01	0	

WORKPIECE COATING VS. SILICON NITRIDE DIE-COMPATIBILITY\*

		<u>GNA</u>	<u>GFE</u>	<u>GCO</u>
1800	Si <sub>3</sub> N <sub>4</sub>	-2.8	-2.2	-2.3
		-0.6	-0.2	-0.3
		-0.5	-0.2	-0.3
		-0.3	0.3	1

\*Table entries denote weight loss encountered by samples during exposure expressed as percent of original sample weight.



TABLE B-III

## ADHESION AND ACCUMULATION

1300°F - Beta C Titanium on IN-100

COATING	Particulate		Applied Load* (Pounds)	Green Coating Thickness (Mils/Side)	Shear Stress* (PSI)	Die Accumulation** (Mils)
	Type	Percent				
22A	-	-	2740	5.0	27.0	2.5
"	TiC	4	2720	5.0	13.2	3.0
"	TiO <sub>2</sub>	4	2500	5.5	20.5	2.5
"	CeO	4	2710	5.2	27.5	1.5
"	BN	4	2680	6.0	19.7	3.5
"	TiC	8	2640	5.1	10.3	2.8
"	TiO <sub>2</sub>	8	2760	5.9	45.6	3.3
"	Ce <sub>2</sub> O <sub>3</sub>	8	2740	5.3	33.2	2.2
"	BN	8	2730	6.0	13.9	4.7
44A	-	-	2860	5.2	8.0	0.5
"	TiC	4	2780	6.0	7.0	1.3
"	TiO <sub>2</sub>	4	2790	5.3	7.6	2.3
"	Ce <sub>2</sub> O <sub>3</sub>	4	2730	6.1	9.4	1.1
"	BN	4	2830	6.2	7.8	0.8
"	TiC	8	2750	6.0	6.8	2.4
"	TiO <sub>2</sub>	8	2730	6.1	7.8	2.3
"	Ce <sub>2</sub> O <sub>3</sub>	8	2720	6.1	14.9	2.6
"	BN	8	2710	6.0	8.6	2.5
32B	-	-	2720	6.0	16.0	1.0
"	TiC	4	2870	5.5	16.4	1.1
"	TiO <sub>2</sub>	4	2800	5.8	17.8	1.8
"	Ce <sub>2</sub> O <sub>3</sub>	4	2675	5.1	16.2	1.6
"	BN	4	2720	6.2	14.7	1.4
"	TiC	8	2720	5.5	18.4	0.4
"	TiO <sub>2</sub>	8	2790	6.1	15.4	1.9
"	Ce <sub>2</sub> O <sub>3</sub>	8	2790	6.0	14.3	2.3
"	BN	8	2780	6.2	7.2	1.9
GNA	-	-	3120	5.9	34.6	-3.8
GCO	-	-	3020	10.0	6.1	NR

Test invalid-  
Beta C alloy  
badly deformed.

\*\*Total of 3 tests

\*Average of 3 tests

TABLE B-III (CONTINUED)  
ADHESION AND ACCUMULATION  
1500°F - Beta C Titanium on IN-100

COATING		Particulate		Applied Load (Pounds)	Green Coating Thickness (Mils/Side)	Shear Stress (PSI)	Die Accumulation (Mils)
Matrix	Type	Percent					
32B	-	-	1470	6.0	26.3	0.6	1.5
"	BN	8			14.5	1.9	
"	TiC	8			24.1	1.5	
22A	-	-	1410	6.0	28.3	1.7	1.4
"	TiC	4			4.3	1.1	
"	TiO <sub>2</sub>	4			25.3	0.8	
"	TiC	8			3.9	1.2	
"	TiO <sub>2</sub>	8			45.2	1.4	
44A	-	-	1410	6.0	26.5	1.4	1.2
"	TiC	8			20.9	1.2	
"	CeO	8			39.9	1.2	



TABLE B-1111 (CONTINUED)

## ADHESION AND ACCUMULATION

1650°F - Ti6Al-4V on IN 100

COATING		Particulate Type	Percent	Applied Load (Pounds)	Green Coating Thickness (Mils/Side)	Shear Stress* (PSI)	Die Accumulation (Mils)
Matrix	Type						
CRT	Mica	5	5	2480	7.4	23.3	1.5
"	Bentonite	5	5	2230	6.4	26.0	1.2
"	CeO <sub>2</sub>	1.5	1.5				
"	+ Graphite	1.5	1.5	3010	64.3	5.7	2.0
"	CeO <sub>2</sub>	5	5	3230	7.5	21.6	0.7
"	Bentonite	5	5	3200	7.1	22.9	1.2
CRT-HA	-	-	-	3400	4.9	19.5	0.3
347M	-	-	-	3180	6.0	41.5	1.7

TABLE B-III (CONTINUED)

ADHESION AND ACCUMULATION1800°F - Ti-6Al4V on Zirconia Coated TRW VIA Dies

WORKPIECE COATING	Particulate		Applied Load (Pounds)	Green Coating Thickness (Mils/Side)	Shear Stress (PSI)	Die Accumulation (Mils)
	Type	Percent				
GFE	TiC	8	1390	6.0	0	-
"	"	"	1420	6.0	16.7	-
"	"	"	1430	6.0	19.6	-

(Coating spalled on cooling after third test - no accumulation data available)

1800°F - Ti-6Al4V on NiAl Coated TRW VIA Dies

GFE	TiC	8	1260	6.0	0	-
"	"	"	1250	6.0	0	-
"	"	"	1420	6.0	0	-
GCO	"	"	1490	6.0	5.4	-
"	"	"	1440	6.0	5.2	-
"	"	"	1430	6.0	5.0	0.3

1800°F - Ti-6Al4V on Siliconized TRW VIA Dies

GFE	TiC	8	1490	6.0	20.0	-
"	"	"	1360	6.0	15.1	-
"	"	"	1360	6.0	16.0	0.4
GCO	TiC	8	1300	6.0	16.5	-
"	"	"	1380	6.0	15.0	-
"	"	"	1370	6.0	13.5	0.2

This table shows individual test results.



TABLE B-III (CONTINUED)

## ADHESION AND ACCUMULATION

1800°F - Ti6Al4V on Silicon Nitride Die

COATING Matrix	Particulate		Applied Load (Pounds)	Green Coating Thickness (Mils/Side)	Shear Stress (PSI)	Die Accumulation (Mils)
	Type	Percent				
GNA	-	-	2770	6.0	21.6	
"			2897	5.8	17.8	
"			Die fractured			
GFE	-	-	1429	6.0	18.6	-
"	-	-	1451	6.0	23.2	-
"	-	-	1403	5.8	18.0	2.5
GCO	-	-	1407	6.1	17.4	
"	-	-	1436	6.0	14.2	
"	-	-	1479	6.2	4.6	
1800°F - Ti6Al4V on TRW VIA Die						
GNA	-	-	1360	6.0	51.6	3.1
GCO	-	-	1400	6.0	31.4	2.2
"	TiC	8	1380	6.0	28.5	1.6
"	TiO <sub>2</sub>	8	1420	6.0	30.7	2.1
"	BN	8	1410	6.0	14.7	1.8
GFE	-	-	1390	6.0	25.8	1.2
"	TiC	8	1380	6.0	26.7	0.8
"	TiO <sub>2</sub>	8	1360	6.0	25.9	1.5
"	BN	8	1350	6.0	5.5	1.3